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PART I

CHAPTER I

RAW MATERIALS

THEIR COMPOSITION AND SIGNIFICANCE OF THEIR COMPONENTS

The raw materials a refiner has to deal with are limited to the sucrose, in the form of raw sugars, extracted from two sources, the sugar beet (*beta vulgaris*) and the sugar cane (*saccharum officinarum*).

Although the sucrose from both of these plants is obtainable as a pure chemical of the formula $C_{12}H_{22}O_{11}$, the raw sugars from either of these sources are very dissimilar, both in composition and refining value. Again, the grades of raw sugar derived from the sugar beet or the sugar cane vary considerably among themselves, even when they are justifiably classed as of the same grade. Thus, centrifugals or crystals, polarization 96, are not always of equal refining worth; nor are those raw beet sugars known as First Products always of approximately the same chemical composition.

Evidently, then, a clear concept of the significance of the constituents in raw sugar other than sucrose is of initial importance to the financial as well as the technical departments of an establishment.

Limiting our immediate inquiry to raw cane sugars and the nature of their non-sugar constituents and the influence these exert on the yield of refined products, the subjoined Table I, may be regarded as an analytical basis illustrating the relative refining value of three standard types, *viz.*, centrifugal, muscovados and molasses sugars. Incidentally the corresponding raw beet sugar products are also given.

It may be observed *en passant* that discussion directed to centrifugals eventually also applies to muscovados and other low-grade sugars as these would practically be converted into centrifugal-like products in the preliminary steps of their refining, and therefore, the major portion of the material handled would be immediately subject to all the conditions obtaining in cen-

TABLE I.—CANE SUGARS

	Centrifs.	Muscovados	Molasses
Polarization	96.00	87.00	88.00
Reducing sugars	0.50	3.50	2.80
Ash	0.50	1.00	2.50
Org. non-sugars	1.80	3.00	3.20
Water	1.20	5.50	3.50

BEET SUGARS

	First product	Second product	Third product
Polarization	96.10	92.10	90.80
Reducing sugars	—	—	—
Ash	0.93	2.07	2.81
Org. non-sugars	1.47	3.27	3.69
Water	1.50	2.56	2.70

trifugal affination. The manipulation of muscovado and molasses sugars will be considered in their appropriate places in subsequent pages.

In commercial analyses and in refinery practice the components of raw sugar may be itemized under six heads:

I. Polarization (approximately Sucrose).
II. Invert Sugar or Reducing Substances.
III. Ash.
IV. Suspended Matter.
V. Organic non-sugars.
VI. Water.

The term reducing sugar will be generally employed in preference to invert sugar, since the former is more embrasive than invert sugar and includes other substances than dextrose and levulose which are fifty fifty components of invert sugar.

With the exception of sucrose and water, none of the other components as designated are definite chemical compounds, and for that reason, according to their varying composition, their scope of influence on the refining yield is more strictly related to their chemical composition than to their mere quantitive presence. A brief review of the significance of each component is here pertinent and necessary so that a fairly comprehensive conception shall enable the reader to follow the particular matter dealt with in this and later chapters.

Polarization: Sucrose.—As polarization, which closely approximates the sucrose content in the higher grades of raw sugar is, of course, the essential constituent of the raw material, it would at first glance appear that raw centrifugals, polarization 96, and the first product raw beet sugar, polarization 96, would yield about the same quantity of, say, granulated sugar. This is not the case however, as it will at once be noted that the beet sugar contains no reducing substances, while the ash percentage is nearly double that of the centrifugal sugar; nor does the one-third per cent lower organic non-sugar balance the higher ash content and the absence of reducing sugar.

Ash.—Analyses of typical ash from raw beet and cane sugars at once reveal the fact that the ratio between the different mineral salts in the ash of the two sugars is of decided importance. Grouping the bases as (I) calcium and magnesium and (II) potassium and sodium, we find a ratio of 1 to 13 and 1 to 4.37 respectively, as shown in Table II.

TABLE II

Sugar	Total ash	Per cent in ash		Ratio
		Ca and Mg	K and Na	
Centrifugal	0.48	8.80	38.50	1 to 4.37
First product	1.03	3.33	43.28	1 to 13.00

For many years it has been empirically known that most of the K and Na salts are powerful deterrents to the crystallization of sucrose from solutions in which these salts are present, and as K and Na salts are found in comparatively large quantities in beet melassa, it is no doubt primarily due to this fact that the melassa of beet sugar origin is so much richer in sucrose than the similar products from the sugar cane. This difference relative to sucrose contents of the respective *last products* is strikingly demonstrated by the fact that beet melassa cannot be obtained at a lower apparent purity than 50° to 55°, while cane molasses, under similar methods, can readily be brought down to 35° to 40°, or even lower. From this it of course follows that, under present practice, less sugar crystals can be obtained from raw sugars in which K and Na salts form a high percentage of the total ash. The importance of these considerations is illustrated very clearly from the data of Table II, showing that nearly two and a half times more K and Na are found in raw beet sugar (first product) than in raw cane sugar, 96 crystals.

Thus:

$$\frac{1.03 \times 0.433}{0.48 \times 0.385} = 2.4.$$

Putting aside, for the present, theoretical discussion as to why one class of salts (K and Na) should inhibit crystallization of sucrose, while another class is neutral or even induces, within certain limits, the growth of sugar crystals, the fact remains that all melassa products of beet origin contain more K and Na, and are uniformly of higher apparent purity, than the corresponding cane sugar products.

Organic Non-Sugar.—Is never determined by direct analysis, but is obtained by subtracting the sum of all the other constituents from one hundred. The organic non-sugar figure, therefore, in commercial analysis includes a large variety of organic compounds in this term.

Numerous, careful and detailed analyses of the organic non-sugars have been made with the result that a great number of organic compounds have been isolated or proved to be present. In a tabulated list of such compounds would be found (1) organic acids, free or combined with the mineral bases of the ash; (2) albuminoid and pectose substances; (3) a number of decomposition products resulting from the juice manipulation in raw sugar production; (4) gums and resins; (5) living organisms and plant tissues; (6) aromatic ethers; (7) coloring matters, etc.

The percentage of organic non-sugar, as a whole, has very little specific value as suggesting this or that organic compound. But as the ash composition of a beet or cane sugar is characteristic of the particular sugar, so also do we find marked and peculiar features in the organic non-sugar compounds of these two classes of raw sugar.

Invert Sugar or Reducing Sugars.—These are always found in comparatively small quantities in the juice of the ripe cane, but may even predominate in the immature canes and in the tender tissues—top nodes—of the fully grown cane. In beet root juices and in the sugar extracted from them, reducing sugars are seldom found except as mere traces, a fact very important to beet sugar production, as it makes alkaline work possible without undue formation of coloring matter, which is so readily formed when reducing sugars are present in alkaline solution under treatment during the manufacturing process. In the case of cane sugar the percentage of reducing sugar is a matter of consideration as first, by their mere presence, reducing sugars induce or facilitate further inversion of sucrose in proportion of the original quantity of reducing sugar present, and secondly, being practically non-crystallizable substances, reducing sugars, if not directly melassegenic, tend to increase the viscosity of the syrups, thus making the crystallization of the sucrose more difficult.

Water.—Water, until recently, was generally believed to be of little importance and was regarded as a simple diluent of the raw material, but investigations into the keeping qualities of raw sugars have brought the significance of this constituent to the fore.

Dr. Browne, of the New York Trades Laboratory, has demonstrated, by a series of interesting experiments, that when the ratio of water to sucrose reaches a certain figure, inversion tends to be set up in the thin film of dilute syrup found on the surface of the sugar crystals. Pure sucrose, as in granulated sugar for example, is somewhat hygroscopic, but when such crystals are coated with a layer of molasses, as is the case in the raw material, the hygroscopic tendency is markedly increased. Therefore, when the humidity of the atmosphere rises, water is absorbed by the raw sugar and the ratio of water to sucrose increases, bringing about favorable conditions for the invertive power of the numerous micro-organisms to assert itself.

Insoluble and Suspended Matter.—The quantity of insoluble matter is never shown in commercial analysis, being included partly in the ash and partly organic non-sugar. Suspended matter merits some attention, however, on the part of the chemist and the superintendent of refinery operations. The quantity of suspended matter varies considerably although its percentage presence is comparatively small. Centrifugal crystals seldom contain more than 0.3 per cent, and in many of the better grades, suspended matter may fall to 0.1 per cent. In molasses and muscovado sugar it may rise to 1 per cent.

The components of suspended matter are (1) sand and clay. (2) sugar-bag fiber and bagacillia, (3) micro-organisms. All of these constituents have a very specific influence on the press and bag-filter operations.

CHAPTER II

VALUATION OF RAW SUGARS

COMMERCIAL VALUATION OF RAW SUGARS (96 BASIS)

Before the advent of the polariscope as a means of rapidly determining the sucrose content of a raw sugar, the raw products submitted to the buyer were judged by a more or less arbitrary estimation of the physical properties of the material, *viz.*, the size of the crystals, the color, the moisture and so on. With the introduction of the inestimably useful polariscope, the valuation of all sugar products as to their sucrose content became a rapid and comparatively definite operation, and though it was soon proved that the polariscopic figure did not necessarily mean percentage of sucrose in the sample tested, the approximation was close enough to satisfy both the purchaser and the vendor. And so "polarization" is to-day the one important figure upon which the commercial transaction is customarily based, at least in so far as raw sugar products are concerned.

But with more accurate determination of the sucrose content of a raw sugar, concomitant closer observation was possible in refinery practice, and it very soon became apparent that non-sucrose substances in the raw material affected the yield in refinery operations. Investigations were made by numerous chemists and refiners to establish certain "Constants" which should be considered as further factors in determining the commercial and refining value of a raw sugar. In short, it was presently observed, and indeed proved, that a definite percentage of ash and organic non-sugars in the raw had a decided and approximately determinable "Melassegenic action." By this phrase was meant that the presence of ash and organic non-sugars in the raw material prevented a certain amount of sucrose from assuming a crystalline form, thereby reducing the yield of white sugar by a definite percentage, and increasing accordingly the amount of melassa or molasses as the case might be. While this was readily admitted to be the case on general principles, the extent of this melassegenic action was not easily established. In fact, no proposed factors as to the melassegenic action of the organic non-sugars, independent of the ash, were ever accepted in the commercial field, and the action of the ash, in regard to raw beet sugar only, was recognized as determinable with sufficient approximation to the truth to warrant considering this factor in price fixing. After much research and lengthy debating pro and con, payments were eventually based upon an accepted factor or factors. Thus a rendement basis of crystallizable sugar in the raw was conceded and raw beet sugars were paid for on a formula illustrated by:

Polarization — (Per cent ash × 3) or, say, polarization 94, ash, 0.90, then,

$94 - (0.90 \times 3) = 94 - 2.70 = 91.30$, Rendement Value.

In this formula we see that 3 is the factor used, but at various times others, ranging from 3 to 6 were accepted and used. However, the factor 3 having been accepted, payment in this case would be made on 91.30 rendement and not on 94 polarization. The rational of this method of calculating the commercial value of beet sugars will become clear after the refining yield of raw sugar, beet or cane, has been discussed.

Commercial Value.—Since payment on raw cane sugar is based on the polarization, irrespective of the nature of non-sugar substances associated with the sucrose, the purchaser is, perforce, obliged to accept, or may receive at times, a raw product of an appreciably low refining value as compared with the commercial value established by the sole criterion of the polariscopic figure. Clearly then, the polariscopic reading cannot be accepted as synonymous with commercial value because the polariscope reading does not necessarily indicate the refining value. More especially is this true in the case of molasses and muscovada sugars. In such instances, as will be pointed out later, "the polarization of a sugar may either correspond to, be greater, or be smaller than the amount of sucrose actually present" because quite a number of substances other than sucrose are optically active in the raw, and, therefore, influence the polariscopic reading sometimes to a considerable extent.

The importance of these facts has been recognized for a long time, but no acceptable remedy has yet been suggested, nor is likely to be adopted in the near future. The only evident recourse of the refiner then is, to select his sugar (whenever choice is possible and a fair sample is obtainable before purchase is made) upon a complete chemical analysis, and then to calculate from this, the refining value based upon the refining experience of his particular establishment, and finally also, take into consideration the nature of the refined products likely to be required. It may then be open to him to choose, say, between two sugars, of equal polariscopic reading but of different composition as regards organic non-sucrose.

Assuming, then, that the selection of the raw sugar be possible and that the only determining factor fixing the price be the polarization, consideration should be given to every item in the analysis as usually made, together with such other data the significance of which the writers have investigated and proved of definite value, not only regarding the desirability of one sugar over another, but also as furnishing information relative to any special treatment a particular sugar may require during refining operations.

These items are:

I. Suspended matter.
II. Micro-organisms; their influence on defecation and filtration.
III. Tintometry, or color analysis.

These points will be taken up under appropriate headings in the discussion of Refining Value, etc.

INTRODUCTORY

Refining Value.—By refining value may be understood that figure which indicates the quantity of a raw sugar—or a definite mixture of sugars—which will produce a required quantity of a standard type of refined products. As has been pointed out, this so estimated refining value has no necessary connection with the commercial value as determined by the polariscope.

The reliability of the calculated refining value depends, essentially of course, on the correctness of the data used. Therefore, as complete a record as possible of the composition of raw sugars refined from time to time should be found in the archives of an establishment. And indeed, where such records exist, together with a clear presentation of the conditions under which the refined products were obtained from the raw material used, a complete basis has been laid for the calculation of a trustworthy refining value.

But similar grades of raw sugar may vary from year to year, affected by all possible exigencies of climate and economic conditions prevailing in the numerous raw sugar producing countries, and therefore the character of the raw products on the market may at any time be a reflection of such conditions. And these conditions—taken in conjunction with the fluctuations of the home market relative to the different demands on one kind of refined products over another—all contribute to raise the proper technical valuation of a raw sugar to a degree of importance not readily appreciated, perhaps, but nevertheless frequently brought home by actual experience.

No better illustration of these contentions can be given than the consideration of reducing sugars present in the raw material. The amount of reducing sugars, may, indeed, be affected by both climatic and economic conditions. In the first instance, meteorological conditions may be such that the sugar-mill receives cane of unusually high reducing sugar content; in the second, economic conditions may necessitate certain lines of extraction; hurried or careless work, lack of proper supervision, all may contribute to the production or accumulation of reducing sugars in the marketed product. And, lastly, if both factors—climatic and economic—of reducing sugar accretion, work in the same direction, this constituent may rise to a threefold percentage over the normal.

Ash and organic non-sugar percentages are in the same category, but not subject, perhaps, to such violent fluctuations, and can be dealt with more readily by adapting refining conditions to the peculiar need of their removal; reducing sugars, however, cannot be eliminated, or to a limited extent only, and must in their proportion contribute to the necessary increased production of last products, *viz.*, either softs or as black strap.

Coloring matters, again, may actually throw twice as much work upon the char battery at one time than at another, and change the nature of the refined products accordingly.

The refiner may not be in a position to alter matters, but in such cases "forewarned is to be forearmed" and a proper and carefully elaborated refining value will contribute useful pre-knowledge of technical problems to be encountered.

The foregoing paragraphs will, no doubt, have suggested that the purpose of establishing refining value is twofold, *viz.;* general and specific, each serving a particular end for which the calculation may have been made.

General Refining Value.—The estimation of a general refining value will necessarily have for its basis some broad line of accepted average efficiency in the refining art, or set upon the data obtained from time to time by a particular establishment. This basis, on the side of the raw material, will rest on definite typical analyses of high grade raws; and on the side of refined products, a standard granulated and a definite last product, will be used to balance the equation.

But as refining implies not only the abstraction of as much sucrose as possible from the raw sugar, but also the elimination of a quantity of non-sucrose substances from the raw material, the quantitative disappearance of this non-sucrose will have to find a proper place in the calculation.

This so-established general refining value, diagramatically tabulated, will permit of a ready approximation being made on the value of a raw sugar under consideration.

Specific Refining Value.—Specific refining value will rest upon quite a similar basis, but as the figure sought will have, as the term indicates, the valuation of some particular raw sugar in view, a calculation will have to be made for each individual case, taking into consideration, also, the relative proportions of refined products required and the amount of char to be employed during the period of refining. However the diagrams referred to on pages 22 and 23 will serve as a means of rapid valuation in most instances.

Method of Calculating General Refining Value.—As has been suggested the general refining value will have for its basis accepted typical analyses of both raw and refined products and the consideration of the elimination of a certain proportion of non-sucrose constituents in the raw. Obviously, then, those types will be chosen which are most commonly used and produced.

Therefore, 96 per cent centrifugals will represent the raw, while granulated, practically 100 per cent sucrose, and a typical black strap will be chosen for refined products. If it should be desirable to also consider the simultaneous production of a certain percentage of softs, this will in no way interfere with the general refining value so long as a constant percentage and analysis of the soft be maintained.

The elimination of non-sucrose substances must also be standardized and considered in accordance with the composition of the raw and refined products accepted as typical.

Taking, then, 96 centrifugals, as the typical raw, we may assume for it the following composition:

Pol.	R. S.	Ash	Ons.	S. M.	Water
96.0	1.00	0.55	1.20	0.25	1.00

For the purpose of illustrating the method of general refining value calculation, let it be assumed that granulated only is to be made together with a necessary amount of black strap, which for the present will be an x quantity.

When the entire raw material to be refined is passed through the char-filters, under the generally accepted quantity of char, *viz.*, 1 pound of char to 1 pound of raw sugar, the adsorption of organic non-sugar and ash is a fairly constant quantity under an established system followed in every refinery; indeed, the only constituents likely to be subject to undue fluctuation will be the reducing sugars and the sucrose. The quantity of the so adsorbed organic non-sugar and ash should not be regarded as lost material, as the greater the adsorption the larger will be the yield of granulated and the smaller the black strap production. Along with the adsorbed organic non-sugar, a certain quantity of sucrose will always be found, and this cannot be profitably recovered in the process of rejecting the organic non-sugar adsorbed by the char. This sugar, then, must be considered as an unavoidable loss of valuable material, which under present refining methods accompanies the reducing sugar, ash, and organic non-sugar eliminated by char-filtration.

Under these considerations, the first step in the determination of refining value, general and specific, will be to subtract from the percentage of the raw sugar constituents such a quantity of each constituent as general experience, or a particular practice, may justify. When full char-filtration is to be the practice, that is to say, when all the raw material is to be passed through a normal quantity of char, it has been found that sucrose loss will range from 1 to 1.5 per cent; that reducing sugars may either be plus or minus by 5 per cent, usually a gain of five per cent; ash will be 20 per cent less in the total refined products, while the organic non-sugar will be 40 per cent less. For convenience of calculation the water may be disregarded or entirely eliminated from the equations, while suspended matter, of course, drops away completely in the first steps of the refinery operations.

Mathematical Data.—Taking, therefore, the raw analysis and the refining losses as indicated, we readily arrive at what may be called Available Raw Material (ARM) by deducting the respective centesimal losses from the percentage presence of the raw sugar constituents, thus:

Raw analysis	Per cent loss	A. R. M.		
Sucrose	96.00	— 1.20 =	94.85	= S
Reducing sugars	1.00	+ 5.00 =	1.05	= 2.20 = R
Ash	0.55	—20.00 =	0.44	
Org. non-sugar	1.20	—40.00 =	0.71	
Susp. matter	0.25			
Water	1.00			
Total Solids	99.00		97.05	= T% Available Total Solids

The analysis taken to represent the raw sugar in this case is merely approximate, and is used for illustrative purposes only.

To facilitate subsequent calculations, and so that the operations may be thrown into convenient algebraical formulae, the available materials are designated thus:

S = Per cent of available sucrose.
R = Per cent of available non-sucrose.

Reducing sugar, ash and organic non-sugar (available) are summed and carried through the calculations as one item, *viz.*, non-sucrose.

T = Per cent of available total solids.

Similarly the refined products obtained from the available raw material are classified under algebraical symbols assigned to their different constituents, thus:

G = Granulated, always 100 per cent sucrose.
g = Per cent pounds granulated on all sugars produced.

Softs.

P = Polarization, or per cent sucrose in softs.
N = Per cent of non-sucrose, *viz.*, reducing sugar, ash and organic non-sugar.
E = Per cent total solids.
d = Per cent pounds of softs on all sugars produced.

Black Strap.

B = Polarization, or per cent sucrose in black strap.
A = Per cent of non-sucrose, *viz.*, reducing sugar, ash and organic non-sugar.
C = Per cent of total solids.

The two unknown quantities are:

y = Weight of raw necessary to produce a certain quantity of granulated sugar.
x = Pounds of black strap that must be made from y pounds raw sugar when granulated or a certain relative quantity of granulated and softs are required.

Finally tabulating the typical analyses of softs and black strap we complete the data necessary for all purposes of determining general refining value.

	Softs		Black strap	
Polarization or sucrose	88.00 = P		35.00 = B	
Reducing sugars	4.00	7.4 = N	20.00	45.0 = A
Ash	1.40		9.00	
Org. non-sucrose	2.00		16.00	
Water	4.60		20.00	
Total solids	95.40 = E		80.00 = C	

The 20 per cent of water in black strap is considered the customary and desirable amount in commercial black strap. This percentage does not necessarily indicate the amount that would be present under working conditions.

Construction of Formulae and Illustration of Method.—From the data now at hand we are able to construct three formulae, expressing the problem in terms of (I) Total Solids, (II) Sucrose, (III) Non-sucrose.

I. In Terms of Total Solids.—As all the solids found in the refined products will have been obtained from the available total solids in the raw, $\frac{T}{100}y$ will represent that quantity in the raw; while, when granulated and black strap only are made, $g + \frac{C}{100}x$ will represent the total solids in the refined products and therefore,

$$g + \frac{C}{100}x = \frac{T}{100}y \text{ or,}$$

$$100\,g + Cx = Ty \text{ or}$$

$$Ty - Cx = 100\,g.$$

II. In Terms of Sucrose.—By analogous reasoning we get the expression in terms of sucrose, thus:

$$g + \frac{B}{100}x = \frac{S}{100}y \text{ or,}$$

$$100\,g + Bx = Sy \text{ or}$$

$$Sy - Bx = 100\,g.$$

III. In Terms of Non-sucrose.—Similarly,

$$\frac{A}{100}x = \frac{R}{100}y \text{ or,}$$

$$Ax = Ry, \text{ or } Ry - Ax = O, \text{ or } x = \frac{Ry}{A}.$$

As we wish to determine the pounds of raw required to produce 100 pounds of granulated sugar and x pounds of black strap, we eliminate x from the formulae I and III.

$$\begin{aligned} Ty - Cx &= 100\ g \\ Ry - Ax &= 0 \\ \hline ATy - Acx &= 100\ Ag \\ CRy - Acx &= 0 \\ \hline ATy - CRy &= 100\ Ag \end{aligned}$$

$$y = \frac{100\ Ag}{AT - CR} \text{ and } 2c = \frac{RY}{A}.$$

In these two simplified equations we have the means of rapidly determining the general refining value of any raw sugar expressed as yielding granulated and black strap, and the different values will indicate the comparative as well as the general refining value of any two or more raw sugars combined.

Arithmetical Calculations.—By substituting the algebraical symbols for their numerical values we get;

$$y = \frac{100 \times 100 \times 45}{(45 \times 97.05) - (80 \times 2.2)} = 107.37$$

$$x = \frac{2.2 \times 107.37}{45} = 5.25.$$

Therefore from 107.37 pounds, 96 centrifugal raw, there should be produced

100 pounds granulated sugar and
5.25 pounds black strap.

One hundred and seven and thirty-seven hundredths is the general refining value of a raw sugar of this type, and the hypothetical refining losses given on page 13 will apply to raw sugar ranging from 94 to 97 polarization.

By deducting the hypothetical refining losses, therefore from the indicated analysis of the raw, corresponding available raw material values are obtained and can be substituted as shown under Arithmetical Calculation.

Specific Refining Value.—Under the fluctuating demands of the market relative to the proportion of the different refined products required, and also to the varying qualities of these, a close approximation of the specific refining value of a raw sugar is frequently to be sought, and so establishes useful data upon which any particular period of refining operations must be conducted in order to obtain results called for in any particular occasion. Usually in such cases a third refined product will have to be introduced into the equation, *viz.*, Soft Sugars. Then the question is to determine how much raw sugar of the particular kind on hand will produce the required proportion of granulated and softs (of a certain quality) and residual black strap, if any.

Assuming, for the purpose of illustration, that the average analysis of softs is to be that indicated on page 14, we will have three expressions: (I) Total solids in the softs, $\frac{E}{100}$ d; (II) sucrose in the softs, $\frac{P}{100}$ d and (III) non-sucrose $\frac{N}{100}$ d. Incorporating these with the respective equations in terms of total solids, sucrose and non-sucrose we get

I. In Terms of Total Solids.—

$$g + \frac{E}{100} d + \frac{C}{100} x = \frac{T}{100} y \text{ or,}$$
$$Ty - Cx = 100\, g + Ed.$$

II. In Terms of Sucrose.—

$$g + \frac{P}{100} d + \frac{B}{100} x = \frac{S}{100} y \text{ or}$$
$$Sy - Bx = 100\, g + Pd.$$

III. In Terms of Non-sucrose.—

$$\frac{N}{100} d + \frac{A}{100} x = \frac{R}{100} y \text{ or}$$
$$Ry - Ax = Nd \text{ or } x = \frac{Ry - Nd}{A}.$$

Eliminating x from equations I and III, as shown on page 15, we get,

$$y = \frac{A(100\, g + Ed) - CNd}{AT - CR} \text{ and}$$
$$x = \frac{Ry - Nd}{A}.$$

These two equations will now be the basis for all questions relative to specific refining value for producing n per cent granulated, n per cent softs and x pounds of black strap.

n = Any percentage on sugar products.

Arithmetical Calculation. Illustrative.—How much raw sugar (Analysis page 13) will be required to make 80 pounds granulated; 20 pounds softs (analysis page 14) and x pounds black strap (analysis page 14).

$$y = \frac{45[(100 \times 80) + (95.4 \times 20)]-(80\times 7.4\times 20)}{(45 \times 97.05) - (80 \times 2.2)} = 103.55$$

$$x = \frac{(2.2 \times 103.55) - (7.4 \times 20)}{45} = 1.77.$$

103.55 pounds raw sugar will therefore yield
80 pounds granulated,
20 pounds softs, polarization 88,
1.77 pounds black strap.

Refining Yield.—Closely related to general refining value and specific refining value, the refining yield serves a particular technical purpose, as, irrespective of direct information relative to commercial values of a raw sugar, the determined refining yield indicates very clearly what results may be expected, or what refined products can be made from a given raw sugar. While general refining value and specific refining value figures indicate the number of, say, pounds of raw material necessary to produce a certain quality and quantity of refined sugars and black strap, the refining yield is based on the question as to what quantity and quality of refined is obtainable from 100 pounds of a given raw.

Not infrequently it may be undesirable to produce any black strap whatsoever, and then it is important to know in what proportion granulated and a certain quality of softs must be turned out to absorb all the available (or, rather in this case all the unremovable) non-sucrose without any residual black strap being made. In such a case, then, the question would be: How much granulated and soft will be produced from a raw sugar to be refined.

General Formulae for Refining Yield Determination.—Let,

x = Yield in pounds of granulated,
y = Yield in pounds of softs,
from 100 pounds raw sugar.

Referring to the types we have been using in all illustrations, the algebraical symbols and their corresponding numerical values will be:

$$
\begin{array}{lcl}
\text{Raw} & = & \left\{\begin{array}{l} S = 94.85 \\ R = 2.2 \\ T = 97.05 \end{array}\right. \\
\text{Granulated} & = & \quad G = 100.00 \\
\text{Softs} & = & \left\{\begin{array}{l} P = 88.00 \\ N = 7.4 \\ E = 95.4 \end{array}\right. \\
\text{Black Strap} & = & \left\{\begin{array}{l} C = 80.00 \\ A = 45.00 \\ B = 35.00 \end{array}\right.
\end{array}
$$

Then again expressing the equations in terms of total solids, sucrose and non-sugar we get:

$$
\begin{array}{rr}
\text{I.} & Gx + Ey = 100\ T. \\
\text{II.} & Gx + Py = 100\ S. \\
\text{III.} & Ny = 100\ R. \\
 & y = \frac{100\ R}{N}.
\end{array}
$$

Substituting $\frac{100\ R}{N}$ for y in formulae I and II we get,

$$
\text{I.}\ G + E\,\frac{100\ R}{N} = 100\ T \text{ or}
$$

$$
GNx = 100\,TN - 100\ ER
$$

$$
x = \frac{100\ TN - 100\ ER}{GN}.
$$

$$
\text{II.}\ Gx + P\,\frac{100\ R}{N} = 100\ S \text{ or}
$$

$$
GNx = 100\ SN - 100\ PR
$$

$$
x = \frac{100\,SN - 100\ PR}{GN}.
$$

But as G always = 100, these formulae simplify to

$$
\text{I.}\quad x = \frac{TN - ER}{N}.
$$

$$\text{II. } x = \frac{SN - PR}{N}.$$

$$\text{III. } y = \frac{100\,R}{N}.$$

By arithmetical substitution of the symbols we get the brief calculations

$$\text{II. } x = \frac{(94.85 \times 7.4) - (88 \times 2.2)}{7.4} = 68.68 \text{ lbs. gran.}$$

$$y = \frac{100 \times 2.2}{7.4} = 29.73 \text{ lbs. softs.}$$

One hundred pounds of raw sugar will therefore yield:

Granulated	68.68 lbs. or 69.70 per cent on sugars produced.	
Softs	29.73 lbs. or 30.30 per cent on sugars produced.	
Total	98.41	

Special Application of Refining Yield.—As was pointed out under the captions Valuation of Raw Sugars (page 7), Refining Value (page 9), raw sugars of identical polarization do not necessarily possess the same refining value nor refining yield.

Demonstration of this fact can be cogently illustrated by applying the refining yield formulae to a specific case.

As a concrete example take two raw sugars (H and L) of identical polarization, *viz.*, 96.22 but of quite different composition as to their reducing sugars, water and organic non-sugar. These two sugars, however, would yield to char adsorption and other operations very similar refining losses, and therefore, by subtracting such losses according to the accepted ratio, we will get very instructive figures bearing on the refining yield of the two lots.

ANALYSES OF SUGARS H AND L

Sugar	Pol.	R. S.	Water	Ash	S. M.	O. N. S
H	96.22	1.45	1.13	0.53	0.21	0.46
L	96.22	0.83	1.25	0.51	0.21	0.78

Subtracting the accepted refining losses we get available raw material:

Sugar	S	R	T
H	95.06	2.22	97.28
L	95.06	1.75	96.81

The refining yield from 100 pounds of these raws would be:

	Sugar H	
Gran.	68.66 pounds	69.6 per cent on sugars produced.
Softs	30.00 pounds	30.4 per cent on sugars produced.
Total	98.66	

	Sugar L	
Gran.	74.24 pounds	75.8 per cent on sugars produced.
Softs	23.46 pounds	24.2 per cent on sugars produced.
Total	97.70	

The specific refining value of the two sugars would be:

H 107.14 to 100 lbs. gran. and 5.30 black strap.

L 106.71 to 100 lbs. gran. and 4.15 black strap.

Anologically the question may arise: How much granulated and black strap can be obtained from 100 pounds raw sugar of known composition?

In this case let,

x = Yield in pounds granulated,

y = Yield in pounds black strap.

Then from 100 pounds raw sugar, polarization 96, (see example page 13), the formulae will then be:

$$Gx + Cy = 100\ T$$

$$Ay = 100\ R$$

$$y = \frac{100\ R}{A}.$$

$$Gx + C\,\frac{100\ R}{A} = 100T$$

$$GAx = 100\ TA - 100\ CR$$

$$x = \frac{100\ TA - 100\ CR}{GA}.$$

But again as granulated is always 100, the two equations may be simplified to

$$x = \frac{TA - CR}{A} \text{ and}$$

$$y = \frac{100\,R}{A}.$$

Arithmetical operations are raw polarization 96.

$$x = \frac{(97.05 \times 45) - (80 \times 2.2)}{45} = 93.14 \text{ lbs. gran.}$$

$$y = \frac{100 \times 2.2}{1.6} = 4.88 \text{ lbs. B. S.}$$

The specific refining value of the two sugars would be

$$H\ \frac{(97.28 \times 45) - (80 \times 2.22)}{45} = 93.42 \text{ lbs. gran.}$$

$$L\ \frac{(96.81 \times 45) - (80 \times 1.75)}{45} = 93.70 \text{ lbs. gran.}$$

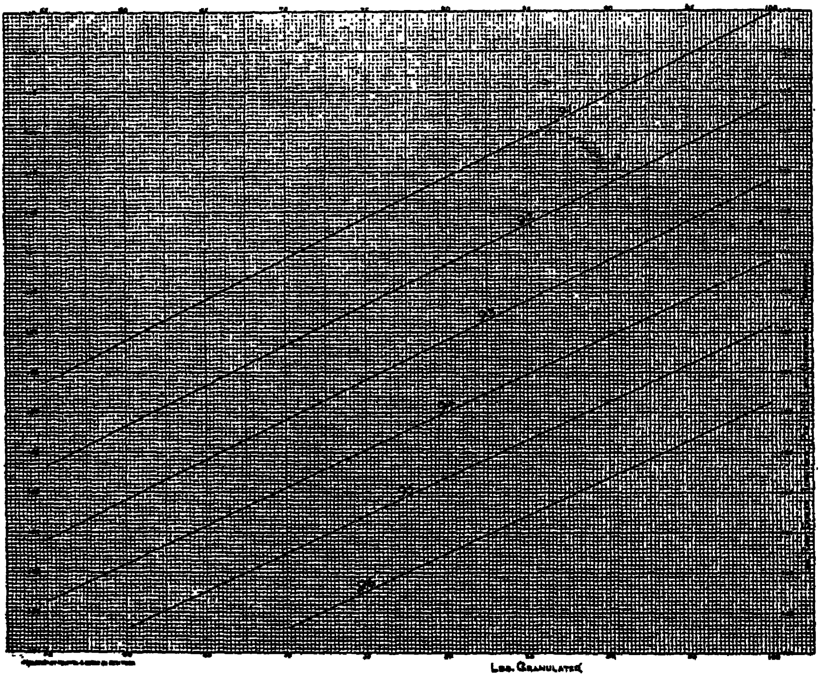

Diagram I.

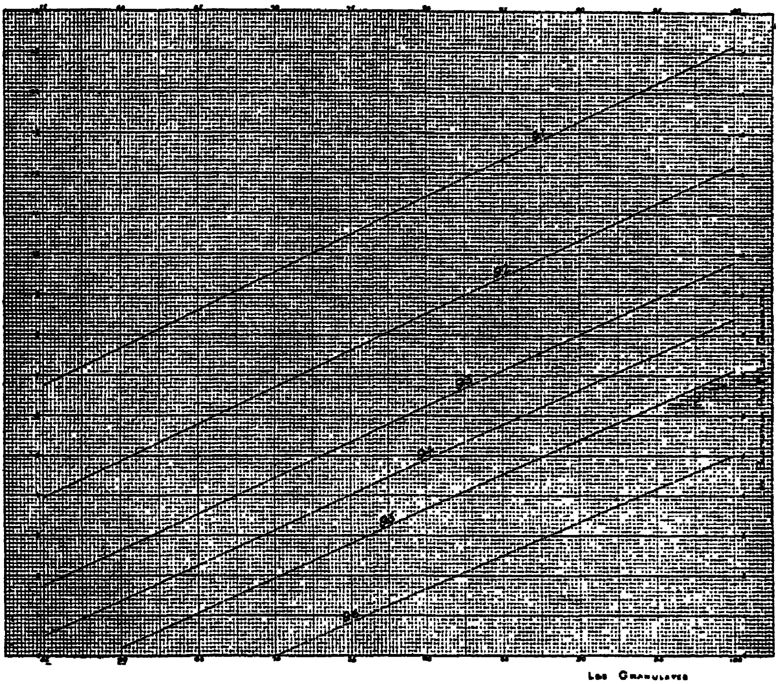

Diagram II.

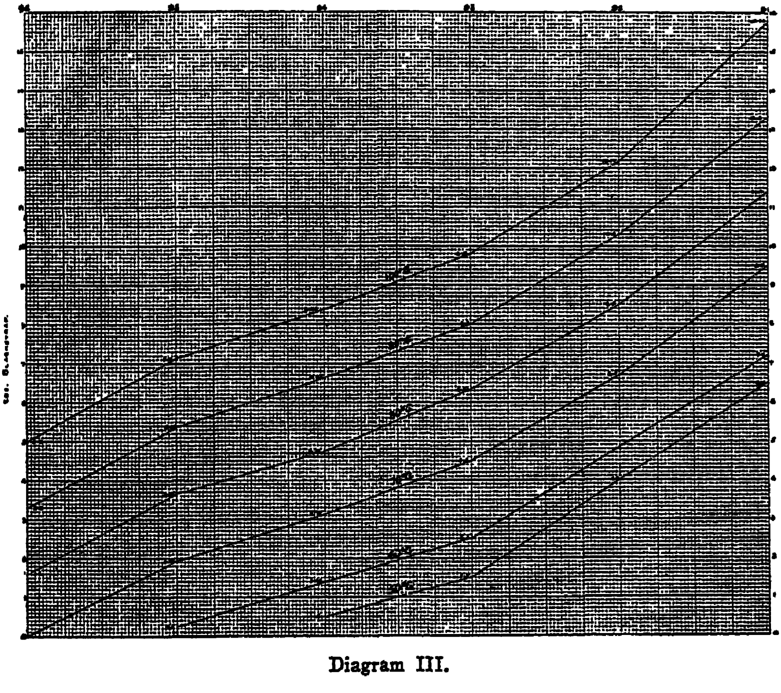

Diagram III.

In conclusion it may be pointed out that all the data so readily obtainable from the calculations illustrated in detail in the preceding pages, should be given careful consideration, not only when the information refers to commercial transactions, but also in the technical operations involving the use to which any particular lot or lots of sugar may have to be applied. More especially is this advisable when a number of similar raw sugars are in store and any particular demand of the market relative to refined products would suggest advantageous refining of one lot over the other.

CHAPTER III

CHEMICAL AND PHYSICAL CHARACTER OF RAW SUGARS

INFLUENCE ON REFINING YIELD AND COST

As has been intimated in the closing paragraph under Commercial Valuation of Raw Sugars, there are important qualities, chemical and physical, in different raw sugars that should receive attention on the part of the refiner. Chemical analyses give no direct information on these points, and, of course, judgment as regards physical characteristics is a matter of observation rather than numerical presentation. Of the five salient qualities influencing the refining yield and cost of any raw sugar, each has its particular factor of importance, but several or all of them may contribute their quota toward a favorable or unfavorable refining result.

Listing these properties in the order in which they approximately enter into refining operations, we have to consider:

I. Water.
II. Acidity.
III. Crystalline Structure.
IV. Insoluble Matter; Organic and Inorganic.
V. Color; Natural and Produced.

Section I. Water.—Although the water content of a raw sugar might be expected, within certain limits, to have no other effect than as a diluent of the sucrose present in the raw product, there are necessarily associated with it other substances which jeopardize the keeping qualities of a sugar very seriously, and these may be regarded as using water as a vehicle for their destructive activities.

Over two hundred years ago, it was recognized that moisture is a prime factor in the deterioration of sugar, and Ligon, as far back as 1673, pointed out the necessity of keeping sugar "drie in good casks that no wet or moist aire come to it." Later, by extending the classical researches and results of Pasteur on micro-organisms to the examination of all kinds of sugar, the basis was laid for a possible progressive and rational study of the deterioration factor of sugars from factory to refinery.

Moreover, it was presently established that active micro-organisms were directly responsible for the diminution of sucrose and the increase of invert sugar in the stored raw material containing more than a certain percentage of water. The reason for this and the correlative phenomena, have been investigated in a masterly manner during recent years by Dr. C. A. Browne of The New York Sugar Trade Laboratory, and his conclusions and practical suggestions are as valuable as his contribution to the purely scientific aspect of the problem bearing on the conservation of stored sugar and the improvement of their keeping qualities by the refiner and manufacturer.

As it appears to the authors, Dr. Browne's work on "The Deterioration of Raw Sugar in Storage," is so conclusive and has so much practical bearing on the initial steps of handling raw sugar at the refinery, that a free summary of his contributions on this subject may form the basis as to the significance of moisture in raw sugar.

Concomitantly, the following paragraphs will equally apply to the advisability of selecting raw sugars, whenever possible with due regard to the points discussed.

In this matter, as in other problems involving a series of factors related to a final result, many empirically derived facts were accumulated before a rational basis was discovered fairly covering the field of enquiry.

Observations on the relative keeping qualities of raw sugars eventually lead to the belief that the ratio between the percentage of water and the non-sucrose in the raw had some bearing on the question, and seemed to indicate that whenever water exceeded non-sucrose to a certain extent, deterioration was prone to occur. Finally a formula, the so-called "factor of safety," was evolved under which it was maintained that, if W be the per cent of water and S be the per cent of sucrose, the quotient $\frac{W}{100 - S}$ must not exceed 0.333. Browne has conclusively shown, however, that 0.333 is too high and that $\frac{W}{100 - S}$ should never exceed 0.3, or better 0.25, in which case the sugar may be considered as of good keeping quality.

The logical basis of this formula is not far to seek now that we have the chemical and mycological data to hand. It is well known that the sugar crystals of even comparatively low-testing raws are chemically pure sucrose, and that all the so-called impurities are present in the form of a molasses film adhering to the crystals. The water present in the raw sugar must therefore be a component of this molasses film which also contains all, or nearly all, of the non-sucrose substances together with a quantity of sucrose soluble in proportion to the water contained in the entire product. But if the crystals are pure sucrose, the molasses film, besides the water-soluble constituents, must also carry the insoluble matters found in the raw sugar, and this insoluble matter includes the micro-organisms, dead or alive, and the spores of such organisms that produce them.

We have, then, in this molasses film the micro-organisms surrounded by all the material upon which they live; some attack the sucrose directly, and others may use the invert sugar for their life functions, *providing that sufficient water be present.* Very few of the organisms are active in undiluted molasses or, more or less pure, saturated sugar solutions, so that if any of the molasses film be kept at a certain density, containing not more than 20 to 25 per cent water, such films will be practically immune to the attack of any host of organisms. This will be the condition of the molasses film on the crystals of a sugar manufactured with due care in this respect, and, indeed, would always be the case if washing the spun sugar in the centrifugal were not practiced in order to bring it up to the required 96 polarization. But if the sugar be washed and insufficiently purged of the dilute molasses formed by the wash water, we have produced, in such a case, the medium and the very conditions for the propagation of any organisms which may be present. But a raw sugar complying with the "factor of safety" at the mill, may be subject to many adverse conditions during transportation and storage before entering the refinery, and deterioration may have been induced several times during the passage from the mill to its final destination. Fortunately our studies of the conditions of induced deterioration are very complete so that proper measures can be taken to avoid losses wherever possible.

Reverting once more to the molasses film we find a full explanation of deterioration by considering what must take place in it as the seat of the entire losses involved. First in order, let it be assumed that we have a sugar with a fair factor of safety, say $\frac{W}{100-S} = 0.3$ but on storage for a length of time, it is noted that the polarization falls, or other signs of deterioration are in evidence; what has happened? Logically, if our data are reliable, water must have been introduced in some way or other, and the density of the molasses film must have been reduced to such an extent that the micro-organisms are able to assume, or reassume their life functions. This is found to be the case; nor need the accretion of water be considerable, since the film may have only its upper surface reduced to a danger density, but nevertheless give ample depth in which the exceedingly minute organisms find a favorable habitat. Indeed, this dilute film of molasses may be regarded as a miniature ocean in which these micro-organisms find all conditions favorable to a prosperous existence—at the expense, very likely of the ultimate possessor of the sugar in which the described phenomena have taken place.

Water, then, is the vehicle through which the direct deteriorating agents, the micro-organisms, are able to exercise their activities. From this follow but two logical lines of endeavor, *viz.*, the avoidance of excess or danger quantity of water, and the exclusion of organisms as much as possible, if keeping sugars are to be made. As regards the former factor of deterioration, the remedy is simple and direct and easily controllable, while the latter involves a general improvement in mill operations and the elimination of all known sources of infection. Obviously, it is impossible to produce sterile raw sugar, but the abandoning of washing centrifugal sugars with water from the cooling-towers —for instance—would at once result in the reduction of organisms to an enormous extent. When we have before us the fact that one gram of raw sugar may contain as few as 3,500 organisms while in another sugar may be found 570,000, we have direct evidence as to the vast improvement possible on many sugar estates.

Without giving a detailed review of all the interesting facts that have enabled the sugar industry to attain a clear and practical concept of the significance of the water content of a sugar as a deteriorating factor, sufficient has been said to emphasize the advisability of taking all possible steps to avoid the now well known conditions that induce deterioration. To the refiner perhaps the most important of these considerations is the storage of the raw sugar. Climatic changes contribute to the bringing about of possible deterioration, even in fairly good keeping sugars if storage conditions are lacking to minimize the effect of such changes. As micro-organisms are dependent upon a certain dilution of the molasses film for their activities, so are they also dependent upon temperature; indeed, where the water content of a sugar is high, or has become so, any rise in temperature will proportionately increase the rate of deterioration if it is likely to take place, more especially if atmospheric humidity should act concomitantly with the first two factors. All raw sugars are somewhat hygroscopic, and when the humidity rises, any exposed sugar, as that next to the bag will absorb water, dilute the molasses film, and if summer temperature prevails we arrive at optimum deteriorating conditions.

APPLICATION OF DETERIORATING DATA[1]

Of course, when the market permits of the selection of one sugar over another, that one should be chosen—with due regard to other qualities—which indicates the highest factor of safety. In the event of the receipt of damaged or unsafe sugars, these should be put through the refinery as soon as possible.

In conclusion the following Table III, based on the formula $\frac{W}{100 - S} = 0.3$, may find occasional application, but too much reliance should not be placed upon this "factor of safety" when the raw sugar polarizes below 95, and, generally speaking, this factor should be considered in connection with grain structure and other physical properties.

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[1] For complete chemical and mycological data see Dr. Browne's article in the *Journal of Industrial and Engineering Chemistry,* 1918, 10, No. 3, 178, also *Louisiana State University Bulletin,* 162, March, 1918.

TABLE III

Sucrose Per cent	Moisture Per cent	Sucrose Per cent	Moisture Per cent
97.50	0.75	92.00	2.40
97.00	0.90	91.00	2.70
96.50	1.05	90.00	3.00
96.00	1.20	89.00	3.00
95.50	1.35	88.00	3.60
95.00	1.50	87.00	3.90
94.50	1.65	86.00	4.20
94.00	1.80	85.00	4.50
93.50	1.95	84.00	4.80
93.00	2.10	82.00	5.40

Section II. Acidity.—The full significance of the acidity of raw sugars bearing on practical refining operations, is a question that has not been given the attention this condition should have received on the part of the chemist. Moreover, acidity is closely related to deterioration as discussed in Section I of this chapter, and should be examined as complementary to the chemical reactions due to the life functions and activities of the micro-organisms that have just passed under brief review.

It may be premised, however, that pronounced acidity presupposes that deterioration is going on or has taken place during transportation or in storage. And for this reason alone, the so affected sugar should be refined as rapidly as possible to avoid further loss.

Section III. Crystalline Structure.—Given two raw sugars, more particularly of the centrifugal type, of quite similar chemical composition, but different as to the size of their "grain," the choice between the two should always lean toward the larger crystal. Indeed, this purely physical difference will influence operations in every department of a refinery and may very materially effect the cost of production.

Apart from the relatively large percentage of sucrose in centrifugal sugars, their economical value lies largely in grain structure, and if this be not fairly satisfactory much of this value will be absorbed by the unusual efforts necessary to keep up to the standard of operation throughout the refinery. In the initial process of washing (affination) the importance of a well-formed and fair-sized crystal becomes apparent. Since affination aims at the maximum removal of the molasses film, spoken of in the preceding section, from the *surface* of the crystals, it naturally follows that the larger the grain the less will be the solvent action of the media of affination, *viz.*, the water finally used to wash the film off the crystals. Again, the degree of perfection to which affination has been carried will determine the facility of subsequent manipulation. Filter-pressing of the dissolved affinated crystals will proceed more expeditiously and the flow through the press will be more ample, lengthening the flow efficiency of the apparatus. Hence will follow less labor of attendance in this department; less washing of press-cloths will mean less water to be evaporated and a consequent economic fuel consumption. Nor is this the only advantage of a freely affinable sugar. When the liquor of such a sugar reaches the bone-black filters, it will contain not more than 1½ per cent of impurities, that is to say, its purity will be 98.5 per cent and the amount of color will also be at its minimum, so that very little work will have to be done upon it by the bone-black and thus the energy of this valuable material will be conserved for the requirements of the syrup filtration. Syrups from a well affinated sugar carry nearly all the coloring matters.

While it is not possible to specifically indicate the pecuniary advantage of one sugar over another in this respect, the disturbance or retardation observed in practice can readily be traced to this patent source of fluctuating refining efficiency.

Section IV. Insoluble Matter.—The influence of insoluble matter on early operations is varied, and, in a measure, related to the affination work as affected by the conditions described in the preceeding section. Generally speaking, fine, ill-defined grain in the raw sugar is accompanied by a comparatively high percentage of insoluble matter, or implies that such matter present offers marked difficulties at the filter-presses. When difficulties are experienced, they may be due to the fact that the insoluble matter is of such a nature that it tenaceously adheres to the crystals even against the passing stream of wash water. This tendency to remain on the surface of the crystals instead of passing away in the washings and syrup, is usually due to the presence of fine argillaceous particles, or, in some cases, to clusters of micro-organisms that have become intimately incorporated with the pitted surface of the crystals. This condition is the direct cause of difficult press work, as both the argillaceous matter and the organisms are in such a minute state of division that they tend to either pass through the filter-cloths and make cloudy press liquor, or being held back, form an impervious and tenaceous skin upon the cloths, necessitating more frequent press renewals, and shortening the life of the filter-cloth.

On the other hand, the coarser insoluble matters are in no manner so objectionable; sand and silicious earthy material are only undesirable in so far that they add to the wear of machinery and injure the press-cloths. Bagacillia, which is sometimes present in comparatively large quantities, has been proved to aid rather than retard press work. Bag fiber may be regarded as extraneous material and cannot rightly be considered as a component of insoluble matter of the raw sugar.

Preknowledge of what working conditions may be expected from any particular lot of raw material can be obtained by experimental laboratory filtration through standard filters and a carefully prepared medium. This should always be done so that a proper estimate may be made of the treatment the lot may require, or, if indications are unusually adverse, to consider the possibility of mixing the lot in question with some other of a more promising feature in this respect.

However, insoluble matter should seldom be a cause for rejecting a sugar that has other qualities to commend it, more particularly if there is any prospect of refining it in conjunction with a more favorable lot.

Section V. Color; Natural and Produced.—The estimation of color, as distinct from color analysis, in all classes of raw sugars has always been considered a matter of importance as instanced by the number of instruments and methods directed towards the determination of its relative quantity. But while such methods were fairly useful in determining the *quantity* of color, they were not at all adapted to even approximately indicating the *quality* of the colors examined; in other words, there was determination of *color depth* but not of *color character.*

It was the recognition of the significance of this condition amongst others, that led to the endeavor by the authors to seek for a more rational as well as a more applicable system to the needs of refinery control in this respect. The Lovibond's Tintometer (inspired by the requirements of the malt industry) seemed to offer possibilities in color analysis for sugar purposes. After a somewhat lengthy period of investigation with Lovibond's instrument and color system, a standard method was finally elaborated which meets all requirements relative to color analysis of raw sugars and has also proved a reliable means of clarifying some hitherto obscure phenomena in refinery practice.

In the Appendix on Tintometry, the technical value of tintometry and its methods will be given in detail, while at present the "color question" will be taken up in so far only as it bears on estimating the relative color types of raw sugars. In a broad way, two color types of raw cane sugar have been recognized, *viz.*, the grey and the red variety. And further, it has come to be generally admitted that grey raws are preferable over the red kind for all purposes of refining; the greys part more readily with their coloring matter than do the red sugars, and therefore, require less char for attaining a certain percentage of decolorization or will yield clearer and more cheerful liquors from equivalent char filtration. But a surprising feature revealed by tintometric analysis is, that a raw sugar in the dry or undissolved state *hides* certain color elements which are discoverable only after the sugar has been dissolved and tintometrized. Again, even in solution the unaided eye cannot detect, within a fair, reliable estimate, the relative presence of the troublesome quantity of red that color analysis so frequently establishes. Indeed, in this fact lies the specific value of tintometry as applied to sugars and emphasizes the statement that character rather than depth of color may be of the greater significance, and be it remembered that measurement of depth of color must be supplemented by analysis before we can have final judgment passed upon the color of any sugar.

In the light of these generalizations, the subjoined Table IV of chemical and tintometric analyses will illustrate a typical grey and a typical red sugar and show what a wide range of effort may be thrown upon the char-filters by one kind of sugar over another.

TABLE IV.—CHEMICAL ANALYSIS

	Pol.	R. R.	Water	Ash	S. M.	O. N. S.
No. 1 Grey	96.20	0.79	0.90	0.46	0.35	1.30
No. 2 Red	96.20	1.59	0.70	0.52	0.27	0.72

COLOR ANALYSIS

	Yellow	Red	Blue	Total
No. 1 Grey	62.0	27.0	5.6	94.6
No. 2 Red	74.0	32.2	2.5	108.7

These two examples are by no means extreme cases, as total color has been known to swing between 60 to 180 total tint. In the example cited, which may be taken as average, No. 2 has only 13 per cent more total color than No. 1. But it is also to be noticed that the two more easily removed colors, Yellow and Blue are 19.4 per cent more and 55.4 per cent less, respectively, or but 11.6 per cent more collectively, while the very undesirable Red in No. 2 (which is exceedingly difficult to remove) exceeds the Red in No. 1 by 19.3 per cent. Here is where the strain upon the char filtration lies.

The Blue color usually disappears completely in the early stages of treatment, and for practical purposes may, generally speaking, be ignored. Where soft sugars are to be made the Yellow color may become an asset. The Red however is the bugbear in char filtration, and it may be said that the percentage increase of red over that in a normal sugar roughly represents the percentage increase in char filtration required to effect decolorization. Thus, if a normal sugar requires 100 pounds of char to 100 pounds of sugar, then a sugar containing 20 per cent more red will require 120 pounds of char to 100 pounds of sugar to secure standard products. Conversely a sugar containing less red coloring matter will require proportionately less char.

This is not the only aspect of red coloring matter, however, for when soft sugars are to be made, the attempt to reduce the objectionable red coloring matter by char filtration results in far greater reduction, almost to elimination, of the desirable yellow coloring matter. In this connection it should also be born in mind that the red color is largely induced by excessive liming, and it not infrequently happens that a raw sugar with an acid reaction, showing a desirable combination of colors in the raw state, assumes a ruddier tint on correction of the acidity with lime. This, however, usually again disappears upon "sulphuring" for the manufacture of softs; while sulphuring of ruddy liquors from red raws, which color has been induced by boiling the heavily limed juices for the manufacture of the raw sugar, by no means responds as readily to the sulphur treatment.

Without anticipating recommendations for the extension of tintometric methods, or discussing here the value of such analyses, tintometry has this happy feature that a reliable color analysis can be made in a short time, and therefore, no doubt need exist as to what may be expected from a sugar about to be bought or of one just arriving at the refinery. Or further, the question of the advisability of mixing lots to some approximation of a standard, will find in the tintometric data the basis for computing what the nature of the mixture shall be, or if, say, a highly colored sugar must be refined by itself, the necessary preparations can be made for obtaining best possible results in such circumstances.

CHAPTER IV

CHEMICAL AND PHYSICAL CHARACTERS OF RAW BEET-SUGAR, MUSCOVADO AND MOLASSES CANE-SUGAR

With the purpose of keeping the subject matter of the preceding chapters in as concurrent a form as possible, nearly all lines of discussion and the illustrative material were based upon typical 96 centrifugal sugar. But the data so established and the conclusions there drawn will now serve as a means of readily passing other kinds of sugar under review, and comparing them in their essential properties with the adopted standard.

Since the better class of raw-beet sugars follow the same preliminary operations in the refinery as do the centrifugal sugars, therefore first and second beet products logically fall into line here.

Similar grain structure and general good affinating qualities begin and end the resemblance between cane crystals and fair quality beet sugar. As has been pointed out, the absence of reducing sugars and the dissimilar ash composition implies that very distinct refining results will be experienced. The most salient difference in the physical appearance of the crystal cane and Product I beet sugar, is the small amount of coloring material in the latter. While cane crystals are seldom under 70° in tintometer standard as adopted in this work, beet sugars (Products I and II) range from 30 to 40, seldom as high as 50° Color analysis of several lots of beet crystals made in 1912, gave the following results:

	Y	R	B	T
Sample I	23.5	7.5	—	31.0
Sample II	26.0	11.0	1.0	38.0

This characteristically low-color content together with the almost entire absence of suspended matter are the two most attractive features in beet-sugar refining. Both press and char-filtration on such sugars can be reduced to a minimum; labor cost in press work will be very low, and char expenditure should be reduced by at least 50 per cent, provided that small quantities of soft sugar be required and a profitable sale can be arranged for the disposal of the unpalatable by-product, melassa or beet molasses.

On the other hand, there are many objectionable features connected with beet sugar refining, and these lie chiefly in the fact that granulated yield is comparatively low; melassa production is high and generally more unprofitable than black strap when there is not a ready outlet to a distillery. Again, when soft sugars are to be made, special precautions must be observed.

If the commercial and refining value of a raw beet sugar be considered, it will at once be apparent that quite distinct factors enter into the case. Taking a typical Product I and referring to pages on Rendement and Refining Value, interesting comparison can be made between cane and beet products in this respect.

In illustration take a Product I sugar as analyzing:

	Sugar	Ref. loss Per cent	A. D. M.	
Pol.	96.00	1.20	94.85	
R. S.	——	0.20	0.20	1.88 = R
Ash	0.95	25.00	0.68	
O. N. S.	01.45	30.00	1.00	
			96.73 = T	

Melassa from beet sugar in comparison with black strap from cane sugar would be:

	Melassa	Black strap
Pol.	54.0	35.0
R. S.	4.0	20.0
Ash	10.0	9.0
O. N. S.	21.0	16.0
Water	20.0	20.0
Total Solids	80.0	80.0
Purity	56.0	43.75

Calculating refining value from these data we get:

$$\frac{100 \times 100 \times 35}{(35 \times 96.73) - (80 \times 1.88)} = 108.18$$

$$\frac{108.18 \times 1.88}{35} = 5.81.$$

In the case of a Product II beet sugar, polarization 92, the difference in the yield factor would be still more marked as appears in Table V.

TABLE V.—RAW REQUIRED TO MAKE 100 POUNDS GRANULATED AND x POUNDS BLACK STRAP OR MELASSA.

Pol.	Beet crystals	Cane crystals	Diff.
96.0	108.18 lbs.	107.23 lbs.	—0.95
92.0	116.43 lbs.	114.69 lbs.	—1.74
	POUNDS BLACK STRAP OR MELASSA.		
96.0	5.81 lbs.	5.00 lbs.	—0.81
92.0	13.00 lbs.	12.17 lbs.	—0.83

Rendement payment on Products I and II in this case would be:

Product I	93.15 per cent sucrose.
Product II	85.17 per cent sucrose.

These data together with comparative cost of the beet against a cane sugar, would form the basis for estimating the commercial value of the raw material. Comparative refining cost is a technical detail, and would form an entirely different problem.

Generally speaking, however, the conjoint refining of raw beet and cane sugars is at times of some advantage, if the factors indicated are kept under consideration. When it is expected that bright soft sugars may be required, a judicious admixture of beet with cane crystals will yield such products at minimum cost.

MUSCOVADO AND MOLASSES SUGARS

These two classes of raw sugar can never be refined to advantage if their admixture with centrifugals exceeds 25 per cent of the total daily quantity taken into the house—and this for obvious reasons.

First in line of consideration is the fact that their grain structure is so small and their molasses content so high that they do not admit of affination and must be melted, filter-pressed and recrystallized to a grain sufficiently large to stand the washing process. These three preliminary steps may easily raise the refining cost from 5 to 10 per cent above that of centrifugal sugar.

Again, their high color content casts a great deal of extra work upon the char-filters, and when the refinery is charged with a capacity production of white sugar, the introduction of molasses or muscovado, not only reduces the relative quantity of white sugar producible, but imposes additional tax upon every department of the establishment. If a comparatively small quantity of soft sugars should be required, and an undue proportion of these lower grade sugars be melted, it follows that black strap will have to be produced in exceptional quantity, and this is proverbially the most unprofitable line of operation.

Typical analysis (page 2) of these sugars has already been given, but these lower products of cane raws vary so considerably that any general commercial or refining value has little purpose, and information of any definite worth must be figured for each individual lot. The same argument applies relative to their tintometric factors; some muscovado sugars may carry three or even four times more total color than a fair quality centrifugal. As an example a muscovado may be cited as typical of high color content. The chemical analysis of the sugar in point was:

Pol.	86.30
R. S.	3.29
Ash	1.48
S. M.	0.47
O. N. S.	2.46
Water	6.00

Color analysis gave:

Yellow	230.0
Red	47.0
Blue	3.0
Total	280.0

An important feature with respect to refining yield must not be lost sight of whenever these sugars, as well as others, come to be regarded in the matter of their stability during refining processes. By this is meant the property of yielding to invertive influences unavoidably encountered at all times. It has already been stated that the mere presence of invert or reducing sugars tend to accelerate further inversion during the time any sugar is subject to heat or any other invert sugar-producing agents. Lack of stability is more marked in low grade raws in which the ratio of reducing sugar to sucrose is high, than in those where the invert content is small, even though other features may not be favorable. Original high acidity or the action of microorganisms can be corrected in one case and arrested in the other, but the influence of a large quantity of original reducing sugar will react upon the liquors and syrups throughout the entire period of production.

Inversion of Muscovado and Molasses Sugars.—In illustration of this fact, two sugars, a centrifugal and a molasses sugar, were dissolved in water, brought to the usual density (56° Brix), made slightly alkaline to the degree necessary in refining, and then the two solutions were subjected to a uniform temperature of 180° F. during 7 successive hours. Determination of the percentage of reducing sugar before and after the strictly comparative treatment and heating brought out the fact that the centrifugal sugar had increased its reducing-sugar content by 24.8 per cent, while the molasses sugar showed an increase of 30.7 per cent. We have it established, then, that a centrifugal sugar of 1 per cent reducing sugars will lose 0.25 per cent of its sucrose under the treatment, while a molasses sugar (polarization 88) containing, say, 3 per cent reducing sugars will lose 1.04 per cent of its sucrose, *i. e.*, four times more than the crystal sugar.

Whatever the cause of this increased inversion may be in the molasses sugar over the crystal, it can be attributed to only two possible factors in the experiment cited. Since any excessive acidity in the molasses sugar was neutralized by the careful addition of lime water, we have only a larger proportion of organic calcium salts and reducing sugars over the other constituents in the molasses as compared with the centrifugal sugar. But it is hardly probable that these comparatively harmless salts should be instrumental in bringing about so much inversion, and it is more logical, it is believed, to attribute the inversion to some form of catalytic action on the part of the reducing sugar, which in such cases may play the rôle of an invertive agent.

When it is of Advantage to Refine Muscovado and Molasses Sugars.—If a certain percentage of the refined product is required as soft sugar of various grades, a properly proportioned addition of muscovado or molasses sugar may be a distinct advantage in several respects.

First, when 10 or 20 per cent of this grade of sugars is refined in conjunction with a fair quality centrifugal, the softs produced can be readily graded; the color and taste will be exceptionably agreeable and the larger percentage of invert sugar in the softs will give the much desired "woolliness" to the products.

Second, other conditions of the work in the house will be favorable to general operations when the quantity taken in is kept within the proportion stated, the muscovado can be melted and the liquor added to the raw syrups from the affination process, thus facilitating press work. And lastly, under the production of softs in larger quantities, the inversion tendencies of these raws need not be feared, since any reasonable amount of reducing sugars formed during the refining will accumulate, without loss of material, in the soft sugars and contribute to the woolly effect just referred to. The apparent slightly greater loss of sucrose can usually be traced to inversion and should not be set down to inefficient work in other respects.

Tables and Diagrams on Yield, Etc.—The following tables and diagrams at the end of Part I will be found useful with reference to some of the material of the preceding chapters, and may be consulted as reliable data for quickly obtaining information as regards refining yield of raw sugars polarizing from 94 to 96.

Arithmetical calculations can be frequently avoided by consulting the diagrams. These give information as to the quantity of a raw, or a mixture of raws, required to yield a desired proportion of granulated and softs, together with the black strap that must be produced.

TABLE VI.—POUNDS RAW SUGAR REQUIRED TO MAKE 100 POUNDS GRANULATED AND x POUNDS BLACK STRAP.

Pol. of raw	Lbs. raw required	Lbs. blk. strap
96	108.2	6.8
95	110.2	8.8
94	112.8	12.3

TABLE VII.—PRODUCING 90 PER CENT GRANULATED AND 10 PER CENT SOFTS, 100 POUNDS RAW WILL MAKE AT

Pol.	Lbs. gran.	Lbs. softs, Pol 88	B. S.
96	84.7	9.4	4.5
95	83.1	9.2	6.3
94	81.3	9.0	9.2

Diagrams I and II can be read in the following manner:

Diagram I.—Follow the vertical line upward, from its junction with the horizontal line (Abscissa) showing per cent of granulated made or required, until it is crossed by any of the oblique lines representing polarization of the raw to be refined. From this intersection move horizontally either to the right or left to the side of the diagram where the marginal figures will indicate the pounds raw sugar required.

Diagram II.—Follow the vertical line in a similar manner to the intersection of one of the oblique lines representing the polarization of the raw, and then move horizontally to the edge of the diagram where marginal figures will indicate the pounds of black strap to be made.

The scope and purpose of the preceding pages will have made it abundantly clear that at the very threshold of refining operations there stand problems and facts representing or creating conditions that must of necessity variously effect the economy of production and influence the nature and quality of the refined material it may be the aim to produce. It has been shown that both the chemical and physical character of the different raw materials are of such significance as to warrant a careful consideration of the manner in which they are known to become factors in the processes of refining.

When the sugar market is free, and competitive sugars are offered for sale, price should not be the only consideration, or, where this is equally placed on different sugars, a thorough acquaintance with all the properties of a particular lot of sugar is always of advantage and the comparatively insignificant cost of analysis and examination may frequently turn the choice from one lot to another. Furthermore, if an unfavorable lot of raw sugar should have to be refined, provisional steps can always be taken for advisable precautionary measures. As matters stand relative to the present manner of buying and standardizing values, it is admitted that many a significant feature of a proffered sugar cannot be, or is not considered under long established custom, but that fact should not mitigate against at least having the facts established, and thus knowing beforehand what the nature of the article may be that is to come before the technical staff for manipulation.

In conclusion it cannot be too strongly urged that typical samples and chemical and physical data of certain lots be prominently kept before the technical staff, and finally, that any one concerned with the purchase of raw sugars should endeavor to get a fair grasp of what the different components of raw sugars signify to the technical management.

PART II

REFINING OPERATIONS

INTRODUCTION

The Chapters of Part I were preparatory in the sense that they marshaled the data which become factors in the initial steps of refinery operations. Typical raw sugars were there passed under review relative to their comparative commercial value, their refining yield, and, finally their physical and chemical properties. The various constituents were dealt with, generally speaking, as integral parts of the raw products, and were disposed of as definite entities for purposes of argument only. But when a raw sugar enters into refining operations, such distinctions largely fall away, and the material in process must be regarded rather as undergoing successive changes as a concrete whole. None of the important non-sucrose constituents of the raw sugar can be eliminated at any one stage of the operations, and many of these persistently follow much of the sucrose into the end products, or are instrumental in destroying sucrose during their elimination.

In Part II, the following listed processes and products will be dealt with in the natural order of the refining cycle, but machinery, reagents, accessory material and control of processes will only be discussed in so far as the immediate needs of the subject may require, reserving for later chapters a more detailed and undisturbed treatment of these matters.

Preliminary.
    Melting.
    Affination.
    Defecation.
    Purification.
    Filtration.
Production.
    Crystallization.
    Centrifuging.
    Granulation.

- Hard and Soft Sugars.
- Fancy Sugars.

Intermediate and End Products.

- Sweet-waters.
- Remelts.
- Final Molasses.
- Table Syrups.

Accessory Materials, Adjunctive Operations.

- Fabrics.
- "Earth."
- Pans and Effects.
- Working Chars.
- Test Papers.
- Indicators.
- Control of Char System.

Theoretically speaking, these processes, typical of all sugar-refining methods, are comparatively simple, but in order that the products shall be of excellent quality and be produced without undue loss of raw material and with the minimum expenditure of labor and energy, much attention to apparently insignificant details is required. While the different steps in the operations are largely mechanical, nevertheless, in a modern refinery chemical supervision is absolutely necessary in order to assure economic production and a standard article for the market.

The first operation in a refinery is determined by the nature of the raw sugar to be refined, if it be a centrifugal sugar, it goes immediately through a washing process (affination); if it be a muscovado or a molasses sugar, the grain will be too fine to admit of washing, and it is therefore dissolved, "melted," and recrystallized in such a manner that the recrystallized product shall closely resemble, in grain and structure, a raw crystal of the centrifugal type and shall be suitable for affination. But be the initial step, melting or affination, modern practice never omits the now very patent advantage of a preliminary washing of a granular raw sugar, followed by the usual steps toward the final goal.

CHAPTER I

PRELIMINARY

Melting.—This is the simplest of all the refining operations and consists of melting the small-grained raws either in water, or more usually, in sweet-waters from different departments of the refinery. Any acidity of the sugar and the sweet-water is corrected by the addition of milk of lime (30° Brix) and the melt kept at a proper density (53° to 55° Brix) at a temperature of 180° F. As in the majority of cases the sugar so melted is destined for recrystallization, it passes out of discussion for the present, but if it should be intended as a mix with the raw syrups it will reappear when these are described.

Hardly any elimination of non-sucrose substances takes place in the melting pan except that the heavy, insoluble material, such as sand, naturally falls to the bottom of a settling tank always provided for this purpose. The milk of lime added, may, however, in some cases not only neutralize the free organic acids, turning them into corresponding calcium salts, but may precipitate some phosphates and organic salts of lime. Generally such precipitation is so slight as to be negligible as a refining factor.

Affination.—Whenever raw sugar possesses a sufficiently large grain, the process known as affination or washing becomes a very convenient method for the removal of the molasses film enveloping the sugar crystals. Obviously this should be done as perfectly as possible with the least solution of the crystals, since these are, as has been shown, practically pure sucrose; in rare cases only are impurities trapped in the body of the crystals, and then, of course, in small amounts only.

Raw sugars of the 96 centrifugal type, having been boiled from concentrated cane juice (called syrup on the plantations) which may have had a purity of from 80 to 88, the grain formed in such a syrup in the vacuum-pan boiling will finally be floating in a molasses ranging from 65 to 70 purity. After this molasses has been purged from the grain, there must then result a raw sugar composed of pure crystals surrounded by an adhesive film of this molasses. We have, therefore, crystals, let us say, 99 per cent pure enveloped by a molasses skin not more than 70 per cent pure. If, then, by some simple means this film of molasses could be separated from the crystals, we would obtain in one step the pure sugar crystals on the one hand and the impure molasses on the other. The means of achieving such a separation are found in a peculiar property of molasses or any low-purity syrup products of the refinery; it is a question of softening the molasses film to such an extent, and also diluting it, that the centrifugal force of the machines will be able to drive a large percentage through the screen, leaving upon it the crystals purified proportionately to the quantity of the molasses ejected.

The softening of the film is brought about by thoroughly mixing 20 to 30 pounds "green" (see below) syrup, raw or refined, with every 100 pounds of raw sugar, thus forming a free plastic mass, called the "magma." In a short time the green syrup becomes thoroughly incorporated with the crystals and forms a homogeneous mixture by the mingling it has received. In the "mingler," a long trough, provided with mixing paddles and ribbon conveyor, the incoming raw sugar meets a stream of the affinating syrup and the syrup and the sugar are carried forward and mixed into a second but larger mingler, called a "mixer" and used as a receptacle for about 100,000 pounds of magma.

Function of the Affinating Syrup.—It has just been stated that affination is to be achieved by a peculior property of molasses or a low-purity refined syrup. This property lies in the fact that a low-purity product, though saturated as to its sucrose dissolving ability, is still able to dissolve the non-sucrose material of which the molasses film is so largely composed. This appears paradoxical, but all low-purity syrups are a paradox in a certain sense relative to the ordinary concepts of the laws of solution. A short explanation in this respect is, therefore necessary here.

Water at a certain temperature will dissolve definite amounts of sugar and no more. At 62° F., water will dissolve a little more than twice its weight of *pure* sugar, sucrose, but when the sucrose is associated with non-sucrose substances in solution, water will then be able to hold these as well, although it may already be saturated as to sugar.

To illustrate:

In accordance with the ratio of solubility of sugar in water, 30 pounds of water at 62° F. will dissolve 60 pounds of sucrose, but a syrup of low purity may have a composition as follows:

Water	15 per cent.
Sucrose	40 per cent.
Non-sucrose	45 per cent.

In this case we have one part of water holding two and two-third parts of sucrose in solution, 33 per cent more than the law of pure solution provides for; in addition to the three parts of non-sucrose simultaneously held. This indicates that the law of pure solution does not apply to impure sugar solutions. It further points to the conclusion that non-sucrose substances can not only intrude themselves upon an already saturated sucrose solution, but can, by such an intrusion, enable the water present to take up more sugar to a varying extent. Conversely then, it is quite conceivable that a saturated sucrose solution mingled with the crystals of a centrifugal sugar will dissolve the molasses film and take up very little, if any, of the sucrose of the crystals themselves. And further, by using such a solution over and over again upon other batches of raw sugar, it must finally, by dissolving non-sucrose, so far fall from its original purity that a final affination syrup will be obtained nearly as low as the molasses film upon the crystals themselves. In actual practice it is possible to lower the purity of the affinating syrup from 10° to 15°, but for a number of reasons it is seldom advisable to force such extreme reductions or to use a syrup for affination purposes lower than 75° purity and 70° Brix.

Affination, then, is the practical application of these recognized facts, and by varying the details of the operation, the composition of the raw liquors and the raw syrups resulting from the preliminary processes may be so modified as to produce the most advantageous conditions which subsequent operations may require.

As the liquors and syrups resulting from this process have to be followed, largely as separate factors, to the final production of all refined sugars, the importance of these preliminary steps becomes manifest and necessitates somewhat detailed elucidation. In this respect a mental picture can best be formed by a diagrammatic presentation of the typical features of the process.

Migration of Materials Under Affination.—By way of illustration:

Let it be assumed that the necessary materials for affination in this case are: (R) raw sugar and (A) affination syrup of the compositions:

	R	A	
Sucrose	95.80	56.0	
O. N. S.	2.80	14.0	
S. M.	0.20	—	Deg. Brix 70.0
Water	1.20	30.0	
	100.00	100.00	
Purity	97.00	80.00	

Again regarding the raw sugar as being composed of crystals of sucrose enveloped in a molasses film, 100 pounds of this material may be represented as containing:

	Crystals	Molasses (RM)
Sucrose	90.60	5.20
O. N. S.	—	2.80
Water	—	1.20
	90.60	9.20
Purity	100.00	65.00

If now 33 pounds of affinating syrup be mixed with 100 pounds of the raw sugar, the resulting 133 pounds of magma will contain:

Sucrose	114.28	=M
O. N. S.	7.42	
S. M.	0.20	
Water	11.10	

Keeping these data in view the migration of the components of M may be readily followed according to the diagram IV.

The net result as illustrated in the diagram is that an affinated sugar of 99 purity has been obtained as against 97 of the original raw, and this raw has been separated from 71.5 per cent of its organic non-sucrose, now found in the green syrup. It should be remarked in passing, however, that although only 71.5 per cent O. N. S. is forced into the greens, at least 90 to 95 per cent of the total color in the raw sugar has found its way into the

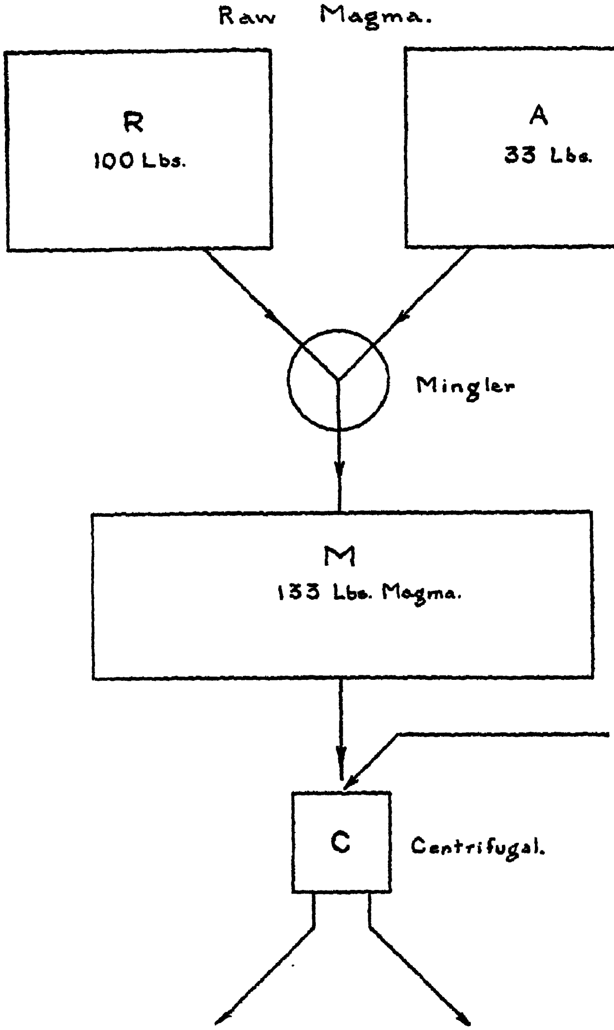

Diagram IV.

green syrup and that the subsequently melted sugar will yield a straw yellow to light brown liquor, imposing very little work upon the char-filtration which can reserve its energy for the filtration of the greens when they come along.

It but remains to explain the compositions of the 53.8 pounds of greens, resulting from the affination. These final 53.8 pounds are derived from the sum of A, RM and W, thus:

	A	+	RM	+	W[2]	=	Gr
Sucrose	18.5	+	5.2	+	8.58	=	32.28
O. N. S.	4.6	+	2.0[3]	+	0.02	=	6.62
Water	9.9	+	1.2	+	3.80	=	14.90

As to a general application of the data it will be presently shown that in practice wide divergences will be experienced, due to the inherent variation of the raw sugars in chemical composition, color content and grain structure; also that consideration of special requirements in other departments of the house may call for modification of affination for purposes of economy or convenience. While, then, changes in detail, but not in principle, are not infrequently effected, the data analysed and reviewed may nevertheless form the basis from which the efficiency of the process may be judged, and, by proper supervision, noted variations can be guided to a desired end.

It now needs no further argument to prove that affination of a raw sugar must be of distinct advantage in the carrying out of subsequent manipulations. Although the process *per se* is simple and the various steps obviously purposeful, there intervene quite a number of factors that tend to, and not infrequently do vitiate the satisfactory execution of the work.

Perhaps the most irremediable factor operating against the production of high-purity washed sugar is found in the grain structure of the raw sugar itself, a fact which has already been touched upon. It is no rare occurrence that along with a fairly developed and uniform major grain is mingled a minor, secondary grain, sufficient in quantity and small enough in size to retard the production of affinated sugar and to lower the purity of it as a first and immediate effect; and secondly there may follow more difficult press-filtration and also a smaller quantity of white liquor from the char-filters. These disturbances are mainly due to the fact that the small grain "clusters" in the interstices of the larger grains when the magma is being purged in the centrifugals, barring the ready outflow of the affinating syrup on the one hand, and on the other, as they offer a much greater surface to the adhesion of the syrup, require more wash water to reasonably free them from that syrup. A partial amelioration of this condition may be found in the dilution of the affination syrup, so, that instead of keeping this syrup at 70° Brix, it may be necessary to lower it to 60° or 50°; in extreme cases water may be the only efficient remedy. But in any case, the use of thin syrups and an abnormal quantity of wash water means that much of the small grain will be dissolved, and in consequence the outflowing greens will not fall but rise in purity, necessitating an eventual reboiling to separate the dissolved sucrose from its associated molasses elements.

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[2] Dissolved by the wash water. If the solids of W be taken from the solids of Gr, it will be found that the purity of Gr would have been reduced from 80.0 to 76.8 if W had not been mixed with it.

[3] Approximately 71.5 per cent of 2.8 per cent O. N. S. in the raw sugar.

Temperature and Viscosity of Affination Syrups.—Another factor in affination work is the temperature of the affination syrup, a consideration which has not been given the attention it merits. The purity and density of the syrups can very easily be kept under control to meet any subsequent requirements, and, given a fair-grained sugar, regulated to a nicety in most cases. But a syrup maintained at quite a satisfactory density and purity will certainly retard the volume of output and reduce efficiency in other respects if it is allowed to become cold, which means that its viscosity increases, its motility is reduced and more energy will be required in all mechanical operations incidental to its movements.

The relative viscosity of a sugar solution is dependent upon three factors; purity, density and temperature, but rise and fall of temperature, more particularly in low-purity solutions, influences viscosity to a much greater degree than either change of density and variation of purity. Affination syrups at customary purity and density are subject to but slight fluctuation in viscosity by reason of their composition, but when the temperature is allowed to wander between 60° and 100° F., viscosity may vary as one to three. Assigning an affination syrup of 75 purity and 70° Brix at 100° F. a unit of viscosity, it has been found that when such a syrup is chilled to 60° F., its viscosity rises to 3.3; that is to say, it is one-third less mobile, will flow three times less rapidly or freely under given conditions than when its temperature is 100° F. Transfer this condition to the interstices of the sugar crystals in the centrifugal and the conclusion as to the advantage of a moderately warm syrup over a cold one is obvious. This applies especially to winter conditions in northern refineries. On cold winter days the incoming crystals may be many degrees below the freezing point of water, and as they are mingled with the syrup, very soon reduce it to 60° F., or lower, if no means be provided for making up for the adsorbed heat. *Moral*, keep the affination syrup at about 100° F.

Pecuniary Return in Good Affination.—While it is quite impossible definitely to determine the economy of good affination work in so many cents per 100 pounds of raw sugar refined, the general results are so evident in this respect that careful and attentive control in this department will always repay the effort expended upon it. Dr. Horn (*J. I. and E. C.*, Oct., 1918, page 810) has made a very fair estimate of the general economy of affination relative to the probable eventual cost of remelts, etc. He says:

"Washing(affinating) an ordinary 96 centrifugal sugar, yields washings of 75 to 80 purity, or thereabouts, and it is highly desirable that all of the impurities possible should be separated from the grain and forced into the syrup, for these two are handled separately, and each additional pound of impurities that hangs back in the washed sugar necessitates just so much extra work for its final elimination. A pound of impurities will lose about 0.4 of its mass in the bone-black, and carry with its remainder about 0.4 pound of sugar into the residual syrup, having a purity of about 40. In washings of 80 purity only about two and one-half boilings or recrystallizations are necessary to effect the separation, whereas in washed sugar of 98 purity, six or seven boilings and crystallizations with all the correlated operations will be necessary to eliminate the impurities. In a washed sugar of 98 purity, therefore, as compared with one of 99 purity, taken as a standard, there will be 1 per cent of the mass which will have to be boiled, etc., four extra times. It follows that each 1 per cent of impurities above the normal 1 per cent left in the washed sugar cost the refiner 1 per cent times four times the cost of boiling, etc., times the price per pound of raw sugar above the basic price of raw sugar, and should be valued accordingly. Conversely, a sugar washing up to 99.5 purity under standard conditions is 0.5 per cent times four times boiling cost times the price per pound of raw sugar less expensive to refine than the normal and should be valued in accordance."

Numerous attempts have been made to devise some method for the determination of the comparative affination value of raw sugar crystals, but so far all of them are open to much adverse criticism in the direction of accuracy and on the score of consuming much laboratory time which may be spent to better advantage in other directions. Indeed, attention to the actual conditions in practice is in this case much more satisfactory, and with sufficient routine tests the best results will follow under judicious guidance of the work as it goes on.

Finally referring to Diagram IV the green syrup from the affination may be turned back to receptacle A to be used two or three times over as an affination syrup, and removed only to the next step in refining process after its purity has fallen to between 75 and 78. If, however, comparatively large percentage of softs be required, there will be no advantage in allowing the purity to fall as low as this, and the syrup may be taken out of the system for defecation at a purity of from 82 to 85. For other reasons, also, low greens are frequently troublesome. Poor defecation or difficult press work may necessitate the higher rather than the lower purity being maintained, as will be pointed out under defecation and filter-pressing.

Defecation.—The diagram and analyses on page 49 illustrate the fact that affination does not eliminate any non-sucrose substances of the raw sugar and syrup, so that the process is simply one of translocation, driving about 80 per cent of the contained impurities into the greens purged from the crystals held back by the screen of the centrifugal. The raw sugar has been separated into two distinct parts, *viz.*, washed sugar from 98 to 99.5 per cent pure and green syrup from 75 to 85 per cent pure, and both are now ready for the next step in refining. The washed sugar is discharged from the machines and dissolved in water or a high purity sweet-water especially set aside for this purpose, to a density of 53° to 54° Brix at 180° F. The greens are diluted with a low-purity sweet-water to a density of from 50° to 53° Brix at 190° F. The so-dissolved sugar and the diluted syrup may be designated Raw Liquor and Raw Syrup respectively, and both hold in solution and in suspension *all* of the original impurities of the raw material from which these solutions have been derived.

Suspensoids.—The process of defecation now to follow aims more especially at the creation of a peculiar condition within the solution of both raw liquor and raw syrup which will facilitate the subsequent removal of all the suspended impurities, many of which are so minute that they may be regarded as suspensoids. Coincidently some soluble impurities are also removed by the defecating agent. The physical appearance of both liquor and syrup will at once suggest the necessity of defecation. The liquor, though light in color, is charged with many minute suspended particles, rendering it turbid, while the syrup, carrying nearly all the impurities, is so dark and muddy as to be almost impermeable to light rays.

The impracticability of any attempt at the direct straining off of the suspensoids in sugar juices and liquors necessitated, no doubt, the original institution of some form of defecation. Ancient arts are replete with devices of this nature, and were empirically used with success before the operators could have had any clear idea of the diminutive masses their various defecants were instrumental in removing from any turbid liquids. It is now known that many of the *larger* suspended particles are not more than one twenty-thousandth of an inch in diameter, and a large percentage of them are a great deal smaller. As individuals these suspensoids will, therefore, pass through any textile material likely to be available for their separation from the liquid in which they may be suspended. Any unaided subsidence is, of course, unthinkable. Again if any persistent attempt at straining off the particles through some very fine textile membrane be resorted to, the membrane will become clogged almost coincidently with the clarification of the flowing liquid. This means that the minute suspensions gradually adhere to the textile tissues, fill up the pores by slow aggregation and finally form a perfectly impervious film of slimy, argillaceous mud on the surface of the cloth and the flow of the turbid liquid stops just about the time the membrane promises to become efficaceous.

Defecants.—The suspensoids of raw liquors and syrups may be regarded as collectively consisting of extremely fine clay particles, micro-organisms, colloidal color and the like. Being so minute the practical elimination of them from large volumes of liquid, cannot be achieved by unaided straining through any ordinary membrane or filtering medium. There must, therefore, be sought some means of creating within the solution a condition to facilitate the clarification of the liquid in hand. A defecant or clarifying agent must be found that will so react upon the suspensoids as to coagulate or entrap the individual suspensoids in the larger masses of which the defecant must eventually be composed. Indeed, a defecant, generally speaking, may be either a comparatively coarse material, granular or fibrous, or a precipitated substance, preferably flocculent, formed in the liquid to be defecated, or added to it as conditions may require but, of course, the defecant must have little or no action upon the chief constituent of the solution—on the sucrose in the case of raw liquors and syrups.

The properties of a defecant likely to be of service for the clarification of a liquid, are dependent upon the nature of the liquid and the number and average size of the suspensoids in it. As regards the treatment of sugar solutions in this respect, many substances have had their day, and some very efficient defecants have gone out of use for reasons other than their fitness as defecants. Bullock's blood was at one time the most popular defecant and was remarkably efficient, supplying by the coagulation of its albumen and fibrin an ideal entrapping material. But phosphoric acid and its soluble salts are now almost exclusively used as the agents for producing a defecating precipitate in liquors and syrups for refining purposes where bag-filters are used. In raw-sugar production and in the beet-sugar industry, lime in the form of calcium carbonate, nascently formed in the juices by the injection of carbonic acid gas (CO_2), is used both as a purifying and defecating agent.

But beside these purely chemical defecants must be mentioned others that may be designated as physical defecants. Although the action of these defecants cannot be regarded as of the same order as the chemical agents, the eventual practical results are quite similar. Among these physical defecants are encountered many substances that have been used, and some still find advocates in certain directions. As examples may be cited sawdust, wood pulp and various disintegrated plant tissues, bagasse for example. Sand, powdered glass and char dust have been tried, and lastly diatomaceous earth, proposed and introduced by Dr. Wiechmann some twenty-five years ago or more, is the favorite defecant of this class.

However, with the exception of diatomaceous earth, now known under such trade names as Filter-cel, Air-cel, etc., physical defecants have historical interest only, and diatomaceous earth will exclusively come under consideration here.

Tricalcium Phosphate, Chemical Defecant.—Tricalcium phosphate may be regarded as a typical example of a chemical defecant. It is practically insoluble in water and neutral sugar solutions, and is formed as a flocculent precipitate by the reaction between milk of lime—$Ca(OH)_2$—and either phosphoric acid—H_3PO_4 or a soluble phosphate, as for instance, superphosphate of lime, $CaH_4(PO_4)_2$, thus:

$$\overset{\text{Superphosphate}}{CaH_4(PO_4)_2} + \overset{\text{Cal. hyd.}}{2\,Ca(OH)_2} = \overset{\text{Trical. phos.}}{Ca_3(PO_4)_2} + \overset{\text{Water}}{4\,H_2O}.$$

Claassen points out that the acid phosphate yields one and one-half times as much precipitate as H_3PO_4, and is therefore preferable, both from a point of view of expense as well as efficiency, for the production of a precipitate in defecation. The following chemical equations illustrate this:

$$3\,CaH_4(PO_4)_2 + 6\,Ca(OH)_2 = 3\,Ca_3(PO_4)_2 + 12\,H_2O.$$
$$4\,H_3PO_4 + 6\,Ca(OH)_2 = 2\,Ca_3(PO_4)_2 + 12\,H_2O.$$

The defecating property of this so-formed tricalcium phosphate is determined by the fact that the precipitate is flocculent, or becomes so after agitation for a few minutes of the hot liquid in which it has been formed. If then, a quantity of this phosphate be stirred into a turbid sugar liquor and a proper quantity of calcium hydrate, *i. e.,* milk of lime, be afterwards added, a reaction, according to the equation above, immediately takes place, and the liquid becomes more turbid than it was originally. But if the liquid be agitated and then allowed to come to rest, the effect of the flocculation of the precipitate will very shortly become manifest. The small aggregations of the precipitate will begin to subside and leave behind a supernatent liquid, clear and brilliant, deprived of all its ultra-microscopically fine suspensoids, which have been safely entangled in the flocculations and have been carried down with them. Defecation has been effected, and it needs but to separate the clear liquid from the muddy deposit by a now possible mechanical filtration through bag-filters.

This process is not applicable to press work as the precipitate forms a slimy cake on the cloths of the press and prevents filtration.

Diatomaceous Earth or Kieselguhr, Physical Defecant.—Diatomaceous earth is composed principally of the silicious skeletons of a minute aquatic plant known generically as diatoms or *diatomaceae*. There is no doubt but that this earth has proved itself the most efficient physical defecant so far put into practice. It owes its peculiar property to the fact that the individual diatoms not only readily mix themselves thoroughly with the suspensoids, but also form a dense, though porous "cake" in the press filters.

Almost any sample of kieselguhr will contain from ten to twelve species of the 10,000 known to diatomologists, and as great diversity of form exists among these organisms, they naturally form aggregations with innumerable and minute interstices so favorable to clarifying filtrations. Any drop of water in which some kieselguhr has been suspended will exhibit this diversity of form and size of diatoms; some are circular, others oblong, linear or variously crescented and sigmoid. But supplementing this form of diversity is the skeletal structure so characteristic of all diatoms. When it is recognized that the largest discoid diatom is but one-fiftieth of an inch in diameter, and that within the perimeter there stretches a reticular tracery of fine silicious threads, leaving trellis-like openings of which one hundred may find accommodation across the diameter, a further defecating property may be easily conceived.

The efficiency of either a chemical or a physical defecant having been once established, its value in practice requires two further considerations, *viz.*, its cost and its press-filtering qualities. Cost is easily established by determining the quantity required for reasonably effective defecation; its filtering qualities is best determined by a trial covering a period in actual practice. Laboratory experiments, though useful when made on a sufficiently large scale and representing conditions as nearly as possible existing in the refinery, are not always conclusive.

Defecants in Practice.—The nature and quantity of the defecant to be added to a unit of either liquor or syrup is determined most conveniently by observing a few trial batches in actual practice; judgment and experience of superintendents may in many cases suggest the proper treatment, or a laboratory test may definitely establish the minimum of the required agents. However, the quantity and nature of the defecants used may be somewhat modified according to the ultimate clarifying method to be adopted, and by the purity of the material defecated. Raw liquors, naturally require less than raw syrups; the former to from 0.2 to 0.3 per cent of the solid matter they contain; the latter two or three times as much as the liquors.

Raw liquors may very effectively be clarified by the addition of kieselguhr alone, more especially when such liquors are to be passed through some form of press-filters. But a judicious mixture of an acid phosphate and lime together with kieselguhr is advisable when bag-filters are to be operated, and raw syrups always flow more freely and clearer filtrates are obtained when a phosphate defacant supplements the kieselguhr. It is to be especially noted any excessive use of a single defecant, or a mixture of two, does not eliminate any trouble that may be experienced in either press or bag-filter work; indeed, excess of the added reagent at all times is to be avoided, and considered as a waste of material and a useless expenditure of energy in more ways than one.

The defecants are added to liquors and syrups, brought to the proper density and temperature in tanks of about 300 to 400 cubic feet capacity and provided with stirrers, or fitted with air agitators. The acidity produced by the acid phosphate is neutralized by the addition of milk of lime, and a further quantity of lime subsequently introduced to bring the treated liquid to an alkalinity sufficiently pronounced to meet the tendencies of acidification during later operations. These acid and alkaline controls will be discussed under refining control methods.

Bag and Press-Filtration.—The defecated raw liquors and syrups having been properly "treated" in the defecating or treating tanks, are ready for bag or press-filtration. The liquors and syrups separated during affination are kept apart and flow in different directions. From the treating tanks they are passed through different sets of bag or press-filters, in which the suspended matter is gathered and the clarified solutions issue.

Two distinct methods for straining off the coagulations, etc., have been in use for a number of years. The older method is known as bag-filtration, and consists of passing the liquors through a number of long cotton bags (24 inches x 70 inches) suspended from the perforated top of a cast iron, or sometimes a wooden, copper-lined chamber of a capacity to contain two to three hundred bags. The construction of these bag-filters provides means for distributing the onflowing liquors in an advantageous manner, and also for receiving and properly dispatching the outflowing clear liquid as it may be required. As each bag-filter is put into use, the first liquors flowing through the bags do not come out clear or brilliant; the tissues of the bags are still too open and the coagulations must have time to partially close them up. After a few minutes' run, however, the liquor flows out free from all suspended particles and it is then allowed to flow to a storage tank from which it is conveyed to other tanks preparatory to its further purification.

Identical results are obtained by the employment of filter-presses of which the Sweetland is at present the most popular in sugar refining. The principle of operating these presses is aptly described thus:

"The principle upon which the Sweetland Filter is operated is very simple, and once the reader fully comprehends the *principle,* the details of manipulating the filter and controlling its operation will be readily understood.

"In old-fashioned filter-presses the solid matter in suspension is deposited in a series of cells or chambers. This makes it necessary to open the several chambers and clean each one individually by hand. In the Sweetland Filter the object is to deposit the solids not in a chamber, but in an even layer *upon the surface* of a plurality of filter-leaves so spaced that adjacent cakes do not touch each other. This reversal of the old principle makes possible two extremely important objects, *viz.*: *Automatic Discharge and Efficient Washing of the Cake.*

"The filter-leaf is merely a circular bag supported upon a suitable structure of heavy wire screen, which prevents collapse of the bag and furnishes free drainage for the clear liquor. An outlet nipple provides the exit for the filter. Obviously if this leaf is supported within a chamber with the nipple passing through the wall of the chamber, and then the chamber filled with liquid under pressure, and the pressure maintained by means of a pump, the clear liquid will pass through the bag and out through the nipple, and as filtration thus progresses the solid matter in the liquid will be deposited on the outer surface of the bag forming an even layer called the cake. Since there is no pressure within the bag and a pressure of, say 40 pounds per square inch against the outer surface, the cake must remain in place on the bag, or leaf, by virtue of the pressure against it."

Many other types of both frame and leaf-presses are being utilized in the sugar industry: full descriptions of which are supplied by the manufacturers.

CHAPTER II

PURIFICATION

EXPLANATORY

The filtration and refiltration of the raw liquors and syrups, the separation of the various filtrates flowing from the groups of filters and the final destination of the filtrates, according to their purity and color, cannot be followed intelligently without the aid of a diagram together with some explanatory matter relative to the general principles upon which the process is based, excluding, however, all reference to detail not immediately pertinent to the operations under review.

Besides the raw liquors and syrups, defecated and clarified during the preliminary operations, there appear in the char-filter house other sugar solutions, which, together with the first mentioned, issue from the char-filters as "filtrates" much changed in chemical composition and physical appearance. As filtrates they are of necessity rechristened and must be followed as such until they are again transformed and known anew in their subsequent functions. In the ordinary routine of practice there are five kinds of liquors to be filtered, and six will appear as issuing filtrates, exclusive of "sweet-water" and "waste-water," which may provisionally be regarded as by-products of the char-filter station.

For the purpose of brevity and ready reference, the various liquors. syrups, filtrates, the different chars and also the filters and tanks will be referred to under appropriate abbreviations, as follows:

VIDE. DIAGRAM V

Raw Liquors as	RL.
Raw Syrups as	RS.
"C" Liquors as	CL.
Refiltering Liquors as	ReF.
Granulated Syrup as	GS.

These appear, after passing through the char-filters, thus:

Filtered Gran. Syrups as	FGS.
RL becomes "B" Liquor as	BL.
RS becomes "C" Liquor as	CL.
Various Filtrates as	AL.

Chars.—There are usually three qualities of char kept for the process, here designated as A, B and C and referred to

A, B and C chars respectively as Ac, Bc and Cc; the filters containing these chars as Af, Bf and Cf.

The running or outflow pipes as ExA, ExB and ExC.

Tk. will stand for tanks.

V. P. will stand for Vacuum Pan.

The authors are accustomed to use a quantity of very pure, refined liquor, technically termed "white" or "settling" liquor, but as this is simply an auxiliary material it will play but a small rôle in the operations to be described.

Char Filters.—The filters are cylindrical vessels, about 9 feet to 12 feet in diameter and 20 to 24 feet high, and of from 1,000 to 2,500 cubic feet capacity. The bottoms are either conical or depressed in such a manner as to allow the insertion of a false bottom in the form of a wooden grating (sometimes of iron) upon which is spread a cotton "blanket," firmly held in place by means of leaden bars placed around the periphery of the filter after the blanket has been laid. The purpose of the blanket is to prevent any fine char grains from being carried into the filtrates.

Tanks.—The tanks (known as pressure tanks) are of any convenient shape and capacity and are set at a height above the filters to give a pressure to the liquors going on to the filters of about 15 to 20 pounds, thus forcing the liquids through the resisting mass of char in the filters and driving it out at the exit pipes.

Tanks and filters are usually covered with some non-conducting material to prevent loss of heat.

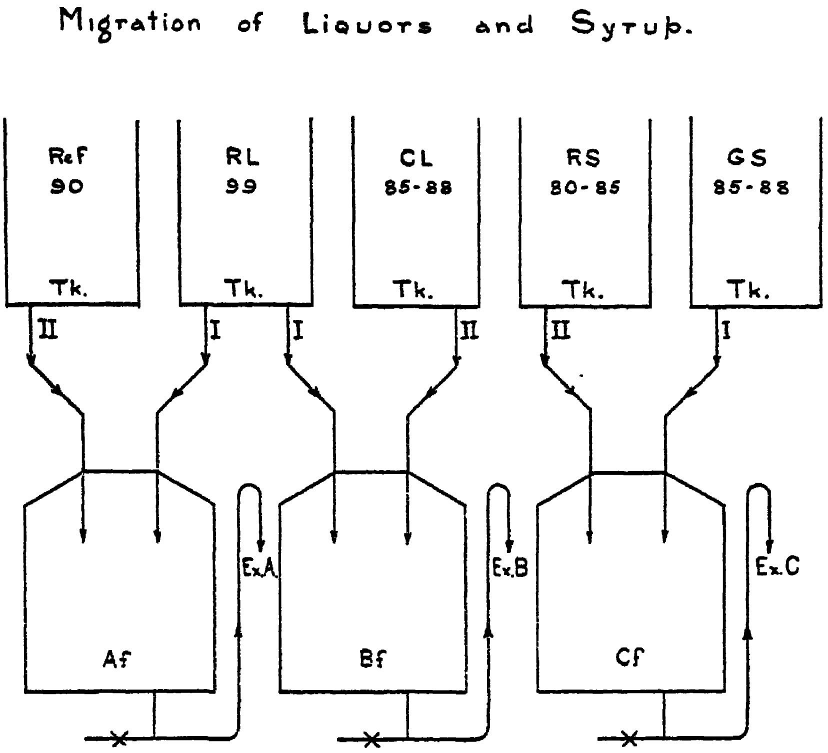


Diagram V.

MIGRATION OF LIQUORS AND SYRUPS

From exit	Flow	Destination of filtrates
ExC	I. GS as FGS	For V. P.
	II. RS as CL	Pumped to CL tank
ExB	I. RL as BL	For V. P.
	II. CL as ReF	Pumped to ReF tank
ExA	I. RL as BL	For V. P.
	II. ReF as	

Roman numerals indicate the order in which liquors and syrups are run upon the filters.

Arabic numerals express the approximate purity of the material in the tanks.

FILTRATION

Order and Method in the Char-filter House.—The first requisite of successful char-filtration is order and method; interruption in the movements of the many tons of material passing in and out of this department, and within it, during the 24 hours of the day, is always accompanied by some falling off from results expected through the steady onward flow of the liquors and syrups and the observance of the once established periodic operations relative to char handling in the filters and kilns, and other incidental requirements. A system of filtration having once been decided upon, it must be adhered to as closely as possible, so that the calculated time for each operation shall not exceed the limit set for it, nor fall below the appointed period, in this respect, the mutually interoperative migrations of the materials is such, that any marked delay on one side or an acceleration on the other results in either congestion or voids as the case may be. The generally accepted practice is such that if 500 tons of raw sugar be refined daily, there would also be used approximately 500 tons of water as a solvent, and another 500 tons of char as an adequate filtering medium, aggregating in the handling of 1,500 tons of material in this department alone. There are then, to be arranged for, a harmonious, coordinated and predetermined volume of liquors and a similarly adjusted supply of char, and means for expeditiously conveying this char to the filters and kilns for its revivification, etc.

Char and Filters.—Char is the most efficient and economic agent known to-day for the separation and final elimination of the non-sucrose sustances from the sucrose contained in raw sugars. Bone-black, animal charcoal, or briefly char, is the final product of the destructive distillation of bone. The charred bone is broken, milled and screened to definite grains, having an extreme range of from 10 to 60-mesh particles, *i. e.*, the individual grains are roughly from one-tenth to one-sixteenth of an inch in diameter. An ideal char in this respect, however, would be one in which all the grains would be of equal size; but as this is not practically possible a range must be chosen, say from 10 to 40, 50 or 60 as determined upon.

As the object of these immediate pages is to illustrate the method and effect of char-filtration, the manner of char activity will not enter into the discussion here. Suffice it to say that impure sugar solutions flowing through a column of char, under certain fixed conditions, leave behind, adsorbed by the char, a portion of their impurities. The sugar solutions issue from contact with this agent considerably raised in purity and deprived of from 90 to 99 per cent of their original color, and contain from 20 to 40 per cent less ash and organic non-sucrose material.

Filling Filters—"Packing."—The char and filters are prepared to meet the best filtering conditions. As regards the filling of the filters, every precaution is taken to insure a uniform distribution of the char, so that a fairly homogeneous column of this material will be found from bottom to top and along all radial lines throughout. To insure this condition, the feeding stream of char—supplying the material at the rate of from 10 to 12 cubic feet per minute—is run through a "distributer" which breaks the main stream of char into a dozen or more lesser streams. Without an apparatus of this kind the single large stream of char, discharging into the filter would form a high central cone above the rising level of the char and the larger grains in it would roll further towards the sides of the filter, thus creating zones of comparatively open spaces. Through such more loosely packed zones of char the liquor would pass more freely than through denser sections and irregular filtration would result in consequence. This condition is avoided by the use of the distributer, by means of which ten or twelve small cones are formed instead of one large one. By the occasional adjustment of the distributer great uniformity is secured throughout the bulk of the char by the time the filter has been filled.

The action of the distributer will readily be understood from the inspection of the sectional sketch.

F is the filter top, C the main stream of the char, c. c. streamlets of the char.

Formerly the packing was done by manual labor. A workman, provided with an air-helmet connected with an air pump, descended into the empty filter and distributed the char coming down through a movable and articulated pipe.

"Settling" or Charging the Filters.—After the filter has been "packed" as described, it will contain from 50,000 to 100,000

pounds of warm char (it should not be more than 120° F.) and is ready to receive any liquor or syrup destined for it.

The general method of charging a filter is thus described by Dr. Wiechmann: (*Sugar*, page 477, 1917).

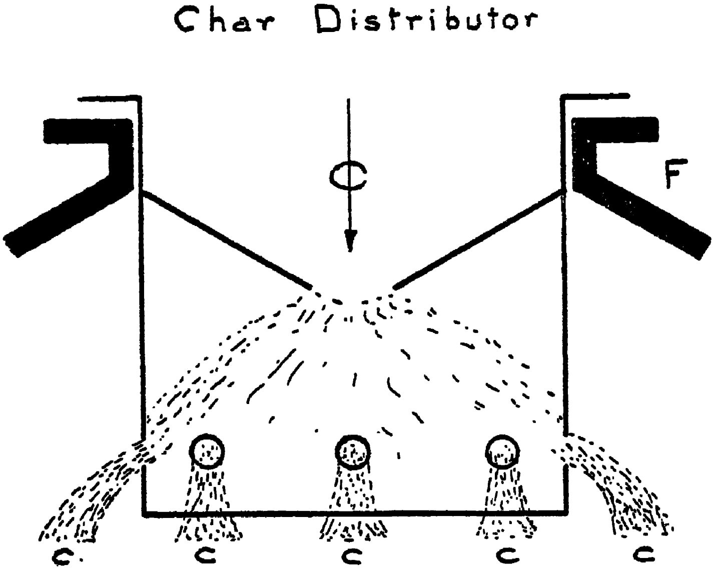


Fig. I.

"When the filter has been properly charged with the black, the sugar solution to be filtered is slowly admitted to the edge of the cone of char at the top. It works its way down the sides of the char until it reaches the bottom of the plate, flowing across this, rises to the top, displacing as it does so the air held in the char. As soon as the liquor appears in the outside delivery pipe its outlet is closed and the filter is then filled more rapidly. When the liquor has reached the top, the inflow is stopped and the filter-man makes sure that all air is expelled; this is readily known by the fact that the liquor is now quiet, that is that it no longer "Boils," as the technical term has it.

"When this stage has been reached and no further settling of the liquor takes place, the filter is thoroughly filled. The filter head is then put in place and the liquor is sent in under a moderate pressure."

This manner of top-settling has several objectional features, which are well worth some effort to avoid. Settling with an open top and on to the upper end of a column of char 18 feet high of necessity requires cautious and slow inflow of the settling liquors to avoid the formation of air pockets in the body of the char. Again the settling liquor, even though run on with the greatest care, *will* form channels, which subsequent treatment may not entirely rectify. Lastly, every moment not used as actual filtering time is so much loss on the total efficiency of the char-filter plant. And it is in this respect that top-settling compares unfavorably with bottom-settling. To adequately top-settle a 1,000 cubic foot filter of char requires from 4 to 6 hours, while bottom-settling can be done in from 3 to 4 hours, and will usually have been done more efficiently.

Bottom-settling.—Bottom-settling requires comparatively little attention and is carried out in the following manner: The packed filter is closed and connection is made at any of the Ex outflow pipes, with a tank containing a store of "white" or "settling liquor." The settling liquor is allowed to enter the filter as rapidly as the char will take it, down through the Ex pipe and in at the bottom of the filter. The liquor consequently rises uniformly throughout the entire column of the char, driving all the air ahead of it and out through an air-cock provided at the head of the filter. The settling liquor having entirely filled the interstices of the char, and also penetrated the cells of the char grains, the filter is now uniformly filled with char, which in its turn is intimately impregnated with liquor and void of any large air bubbles; it is ready for normal filtration at once and may be expected to give no trouble due to careless and hurried settling. When actual filtration begins the inflowing liquor "presses off" the white liquor which is sent to the pans and later a sufficient quantity of new white liquor is collected to settle another filter.

The now packed and settled filters will be set aside for awhile, in order to describe the system of char utilization in vogue.

Char Utilization.—For many years after the remarkable properties of bone-black had been recognized, the black was used in powdered form, in which state, it may be remarked, it is most active, and was added to the liquid upon which it was to exert its powers.

This was the procedure commonly followed in the sugar industry, so that the char in the form of a fine powder acted both as a defecant and decolorant. Nor was any revivification attempted; the once-used powder was rejected as of no further value as a decolorizing and purifying agent. Of course, the advent of granular char together with the discovered fact that by various chemical treatments, washings and heat revivification, its original powers could be largely restored, was an advance of immense importance to many industries which had hitherto been able to use char in very limited quantities only. Filtration with granular char began at this period, and the treatment of used chars with the object of their revivification became an art in itself.

System of Utilization.—The purifying effect of a char is dependent upon four factors, time, temperature, quality and quantity.

Time.—By time is understood the period of contact between a sugar solution and the char in the filter; in other words, the rate of flow determines the time a particular batch of char is allowed to react upon the solution to be filtered.

Temperature.—The purifying effect of a char increases with the temperature at which filtration is conducted, but the practical limit of temperature is set by the nature of the solution to be filtered.

In refinery practice it ranges between 170° to 185° F.

Quality.—Starting with a normal, virgin char, successive filtrations and revivifications bring about such chemical and physical changes in the char grains that its power of abstracting impurities from solutions definitely diminishes with each cycle of use, so that if the operations be persisted in, the so-used char will eventually lose its original powers altogether. Its adsorption power is therefore a measure of its quality, and the more frequently a char has been used the less work will a unit quantity perform, time, temperature and quantity being constant.

Quantity.—The quantity of char reacting upon a unit of liquor solution is directly proportionate to and, together with the other factors, determines the total adsorption obtained.

Utilization in Practice.—Adapting the foregoing data to a practical system of filtration, provision should be made to the end that the filtration of all the sugar solutions of the refinery shall pass through a quantity of char of three qualities, to be known as A, B and C char: A the best, B the medium and C the poorest quality. Each of these chars should have a complete system of filters, kilns and conveyors, etc., by which all operations incidental to their function can be performed without any intermingling and the standard of each char can be maintained. The relative proportions in which Ac, Bc and Cc are used may be varied as occasion requires, but about an equal quantity of each is usually employed.

For example, if 300 tons of raw sugar were the daily "melt," 100 tons of each of the chars would be placed at the service of the char-house, and the standard of quality would be maintained by the daily addition of from 0.50 to 1 per cent of new char on the daily melt according to the quality of the incoming raw sugar and the refined products to be made. As a certain amount of char dust is formed by attrition due to the movement of the chars, this dust is constantly removed and taken out of the system, being replaced by the new char added. Ac is used to replenish Bc and Bc to replenish Cc.

Assuming now that Ac, Bc and Cc systems are working and the Af, Bf and Cf (vide Diagram V.) have been packed and that Af and Bf have been settled with white liquor as described, filtration may be set in operation. Cf is usually top-settled and therefore no white liquor used in connection with it. Cc being the poorest char receives the low purity syrups for a preliminary filtration.

Referring again to Diagram V, page 65, we have the three filters Af, Bf and Cf ready for use; the liquors and syrups, RL and Rs, are constantly entering the system by being pumped from the preliminary house—technically termed "Raw-house"—into the tanks RL and RS.

A Typical System of Filtration.—Tanks RL and Rs having been filled, char filtration is started.

RL being of high purity (99) and containing little color can usually be almost completely purified and decolorized by passing through Bf at the rate of about 100 cubic feet per hour, or from 4 to 6 hours' contact with the char. Indeed, a large portion of the RL issues at ExB as BL and so long as this happens the BL filtrate is run to the V. P. to be boiled.

RS receives first of all a preliminary filtration through Cf containing the poorest char. By this means the Cc is made to adsorb impurities to its limit and thus relieves the better chars of some of the hardest work, conserving their energies and enabling their greater powers to be exerted upon the substances that yield only to the most energetic chars. RS flows from ExC as CL, and is pumped to tk. CL prior to a refiltration.

But the Cc in Cf is frequently used for another purpose before RS is run upon it. From the boiling of granulated sugar there result many cubic feet of syrup too low in purity and too dark in color to admit of further boiling for granulated. These syrups may, however, be very materially improved by passing them over a char-filter, and the Cf offers a convenient route and means in this direction. When the GS is to be filtered it is pumped to the Gs tk., reduced to the proper density, raised to the proper temperature and a definite volume run upon Cf before the RS, which in its turn follows. When a Cf has thus been utilized the GS flows off a ExC as FGS and is returned to the V. P. A certain amount of mingling takes place at the junction of the GS and BS in the filter, resulting in a mixture of the two. But as the CL is invariably much darker than the GFS, the mixture at once becomes apparent, and as soon as it shows the filterman changes the direction of the flow from the FGS gutter to the CL gutter.

Reverting, now, to the Bf and considering that it has received its quota of RL, this filter now receives the CL from the Cf. The large volume of RL has by no means exhausted the Bc and it is still able to do a considerable amount of work, hence it is burdened with the task of removing more impurities from the still dark and low-purity CL. Here again we observe the same mingling of the BL and CL in the filter, and the same means are employed to prevent any of the darker liquor from passing into the light colored BL; the filter-man changes the BL into the ReF as soon as the former shows signs of darkening. The entire volume of CL that went into Bf, and some of the RL intermingled with it, flows from ExB as ReF and is pumped into the ReF tank, ready to receive its final filtration through Ac in Af. ReF is still dark and somewhat crude in color and retains much of the coloring matters that only the most active char will adsorb, and as Ac has that quality, it is given this task to perform. Not infrequently the volume of RL is so large, that for the relief of the Bf some RL may be run on the AF, ahead of the ReF. In any case, the Ac is so active that a quantity of almost white liquor is obtained from these filters, and as the purity is also high enough, the first flow from ExA is run into the BL coming from ExB. Again, when the outflow from ExA becomes tinged with yellow, or the purity falls below a chosen standard, the stream from ExA is run by itself and is known as AL.

Separation of Char Filters.—While the color demand of a liquor is inexorable if a certain quality of refined sugar be required from it, purity is also a consideration, of course. The three kinds of liquors flowing from the char filters are therefore graded according to their color and purity, and are conveniently referred to a B liquor (BL), A liquor (AL) and Filtered Granulated Syrup (FGS).

But under certain conditions, AL and FGS may disappear as such and be absorbed by the BL. Or again FGS may also disappear, passing into AL, if composition and color be right. Obviously the BL is of the highest purity and carries the least amount of color, and color and purity are largely under control in this liquor. AL and FGS, however, are not so completely adjustable in this respect, the color particularly is likely to vary considerably. Since, upon last analysis, AL is the filtrate from the RS, which carried 90 per cent of the original color in the raw sugar melted, the depth of the residual color in the AL will be determined to a great extent by the quantity and nature of the color in the raw sugar. As has already been pointed out, exigencies may arise which necessitate the refining of raw sugars carrying, three, or even four times more color than is normally expected. Naturally under high color content in the raw the concentration of unadsorbable color will be found in the lowest purity filtrates flowing from the char-filters.

Limits of Adsorption.—But the adsorption powers of the best chars are limited and, indeed, begin to decline before complete exhaustion has been reached. In practice it is not advisable to stop the addition of liquors to a filter the moment it falls from its maximum efficiency, nor is it of advantage to carry on filtration until exhaustion has been fully established. A medium course is therefore adhered to, and the filter is given such quantities of RL and RS as the actual refining conditions require or circumstances make imperative. In this respect, progressive exhaustion can readily be observed by comparing samples of filtrates taken from time to time with those of an earlier flow, and also by determination of the purity of these samples.

Since char adsorbs, color, ash and organic non-sugar, exhaustion is indicated by increasing color and by decreasing purity in successive samples of the filtrates.

This is strikingly illustrated by following the course of filtration relative to purity, density and color of a liquor of known composition running through a single filter. If samples be taken at stated periods and the purity and color determined, the progressive changes in the composition of the filtrate will be a fair indication of the gradual exhaustion of the working char. On page 65 will be found a tabulation of such purities and densities of a typical Ac filter. Meanwhile referring to Table VIII, relative to separation of char liquors, it will become evident that batches of liquors or syrups can be obtained at any required purity by running fractions of these liquors into separate vessels. Note also that in this specimen filtration, the ReF has an unusually high purity, *viz.*, 93. If this purity had been, say, 85 or 88, all the individual tests would have been correspondingly lower, but the falling off of the purity, as filtration progresses, would have been approximately of the same degree. It is quite within the scope of practice that filtrates, suitable for boiling, may flow from the filters having a range of purity 80 to 100, so that, by proper fractional separation, liquors could be obtained anywhere within the degree of purity indicated.

Sweet-waters (SW) and Waste-waters (WW).—The rôle of sweet and waste-waters in refining operations will be taken up here in so far only as is necessary to complete the description of the filtration cycle. After a char-filter has had its quantity of RL or RS run upon the char and has discharged its filtrate as BL or CL, the filter contains 600 to 1,000 cubic feet of RL or RS as the case may be. This 600 or 1,000 cubic feet, in fact, is part of the normal quantity of liquor the filter was expected to handle, and the next step is to drive the residual liquor out of the filter. With this end in view, filtration is suspended for awhile, the liquor in the filter is allowed to settle to the level of the char and connections are changed so that hot water (about 200° F.) may be run into the filter, at a much reduced rate of flow as compared with the filtration of the liquor. Instead of running at the rate of 80 to 100 cubic feet per hour, the flow is reduced to from 40 to 60 cubic feet. This is necessary, since the inflowing water pushes the heavier liquor or syrup ahead of it; but the retreating liquor adheres to the char, so that by this adherence, together with the diffusion of the liquor into the water above it, a quantity of dilute filtrate is formed, known as "sweet-water." If the original rate of flow were kept up, larger quantities of liquor would be left behind to mingle with the water and consequently a larger quantity of SW would be formed in the filter, which is to be avoided for several important reasons. But this SW becomes progressively thinner and thinner, its density falls and so also does the purity. Referring again to Table VIII, the letters SW will be found opposite the Br. 46.8, which means that at this point no further benefit could be derived from the filtrate, since it will be noted that the purity has fallen to 93.3, very little higher than the original ReF run upon the filter. Obviously to continue running such a filtrate to the V. P. for immediate use would mean that the filter would be delivering liquor of no higher purity than the ReF, and consequently no purification would be achieved at this point. All the filtrates, then, running between the twenty-second and thirty-second hour will be gathered in a separate tank and dealt with for recovery of the sugar they contain.

Waste-water (WW).—Again opposite the Brix 0.4 the letters WW indicate that the filtrate (known as waste-water) is run off to waste, turned to the sewer as the sucrose in it is no longer worth recovery, if for no other reason than that the cost of evaporating would be more than the sugar is worth.

As soon as the filtrate is changed to WW, the flow is increased to about 200 cubic feet per hour in order to complete the washing as rapidly as possible, so that within 5 or 6 hours the char has been washed; the water having dissolved out a large percentage of the substances adsorbed during filtration.

Filter Cycle.—This completes the cycle of operation as regards filtration, but the complete cycle embraces, of course, other steps before the same filter can again become useful in its filtering capacity.

A complete cycle of a filter consists of the following:

I.	Packing	1 to 2 hours.
II.	Settling	3 to 4 hours.
III.	Running RL	30 to 40 hours.
IV.	Running CL	10 to 20 hours.
V.	Water	20 to 24 hours.
VI.	Pumping	5 to 6 hours.
VII.	Emptying	3 to 4 hours.

A complete cycle will therefore keep a filter engaged from 70 to 100 hours.

Details on SW, WW, Pumping, etc., will be treated fully under Adjunctive Operations.

CHAPTER III

PRODUCTION

EXPLANATORY

The liquors BL and AL flowing from the char-house into the "refinery," a building devoted to nearly all operations mediate to the final processes of production, are gathered according to their purity and color in a series of tanks, prior to their distribution for eventual boiling in the vacuum pan. As was the case in the char-house so in this house, the material entering it at a certain stage in the general process, leaves a step further advanced. In the char-house the raw liquors and syrups were received as comparatively impure, dark solutions, but were sent from it highly decolorized and of much greater purity. This char-house product enters the refinery as solutions of varying quality, but leaves it as a crystalline substance practically ready for the consumer.

On page 78, the successive operations may be followed diagrammatically, apparatus and machinery being lettered thus:

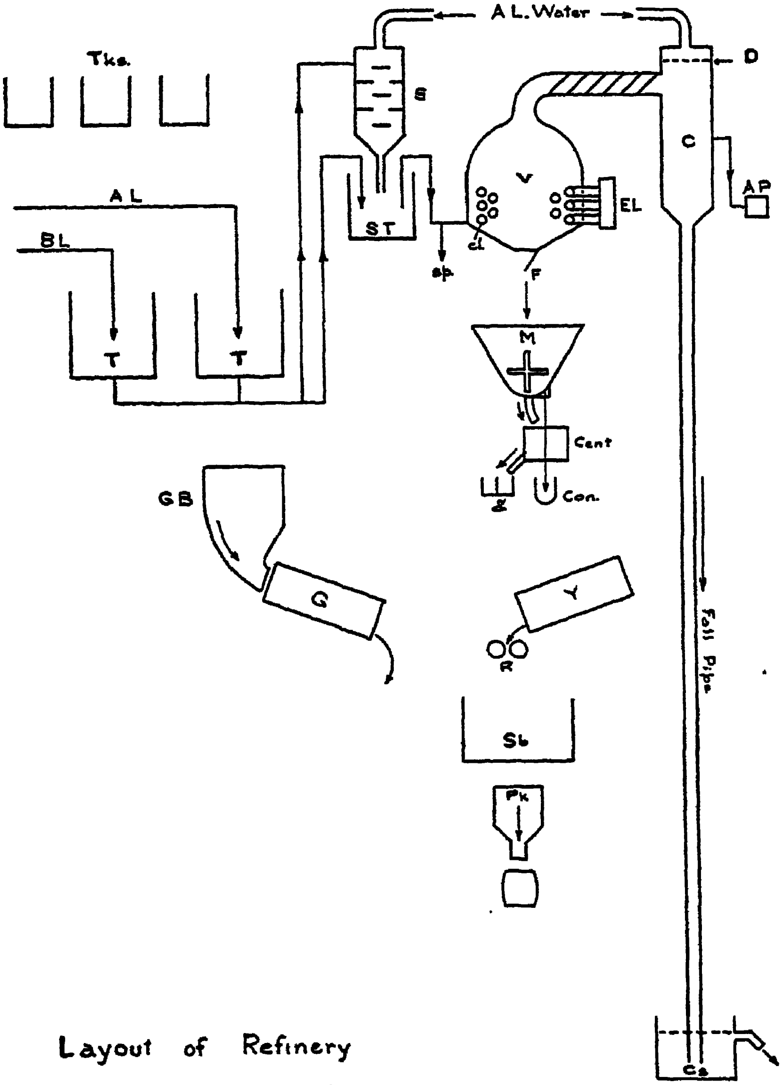
V. Vacuum Pan.
F. Foot-valve.
C. Condensor.
D. Perforated Diaphragm.
Cs. Seal Tank.
AP. Air-pump.
cl. Steam Coils.
sp. Suction pipe.
ST. Suction Tank.
S. Sulphur Tub.
T. Tanks.

M. Mixer.
g. Syrup Gutters.
Cent. Centrifugal.
con. Conveyor.
gb. Granulated Sugar Bins.
G. Granulator.
Y. Yellow Sugar Bins.
R. Rollers.
Pk. Packer.
EL. Exhaust or Live Steam.

Distribution of Liquors.—As the BL and AL liquors from the char-house accumulate in their respective tanks, their purity is determined and their color noted, and, according to these qualities and the nature of the product required, they are prepared and distributed as occasion calls for. The purity and color of

BL fits it for boiling to granulated; the AL varies much in color and purity so that each tankful must be assigned to proceed either by itself or be mixed, in determined volume with BL to make a mixture of a quality required.

"Hard" and "Soft" Sugars.—"Hard" and "Soft" are the generic or class names for the two most distinctive kinds of refined



Layout of Refinery

Diagram VI.

sugars made, and as the process for the production of each is somewhat different, and as the softs may be regarded as the after-products of the hard sugars, the boiling and production of hard sugars will receive first consideration.

The hard sugars include the differently grained, granulated and special products made from such grain.

Boiling to "Grain;" Granulated Sugar.—The B liquor collected in tanks requires no further treatment and is ready to be boiled to grain for the production of granulated sugar. The liquor is therefore pumped from T (v. Diagram VI) to ST in quantity demanded by the capacity of V, which is made ready for the boiling as follows: All valves of pipes leading to the body of V are closed and F securely locked, thus shutting off the two bodies, V and C from the outer air; Cs containing enough water to fill the fall-pipe between the bottom of C and Cs. AP is now started and water is turned on to C and falls through D in numerous streams down the fall-pipe into Cs and thence to the sewer. By this means a vacuous space is rapidly created in pan and condenser and the apparatus is soon ready to "draw in" the liquor in ST into V by way of sp. When V has been half-filled with liquor the valve in sp is shut, and exhaust or live steam from EL is turned on, flowing into the system cl, the liquor soon begins to boil at a low temperature (110° to 120° F.) due to the vacuum maintained by the AP and the cold water condensing the vapors as they rise from the boiling liquors. Evaporation goes on apace until minute crystals begin to form in the boiling mass. Grain formation has begun; crystallization is in progress.

THE SCIENCE AND ART OF CRYSTALLIZATION

The laws governing the formation of crystals from solutions containing a substance that can be crystallized are well known, as regards commercial products at least, but the conditions obtaining in practice are so varied and change so rapidly that any attempt at systematic application of these laws has been proven utterly vain. Crystallization or "grain-building," in quantity and with the rapidity required in industrial operations, is rather an art than a science. While crystallization in practice goes on, of course, under the "reign of law," the prevailing conditions during the process are so evanescent that quick judgment born of experience is the best fitted to maintain the necessary equilibrium in the constantly changing mass under observation. Perhaps in no other crystallization process is this more pronounced than in sugar boiling as now practiced. Not alone are crystals required that shall be free from imperfections due to untoward conditions at birth and during growth, but the finished crystal is frequently expected to approximate a specified size and be characterized by normal development. For these and other reasons sugar boiling must be considered preëminently an art at which science assists as the guiding spirit only.

The density or concentration at which the liquor in the pan begins to develop crystals, is known as the "critical density," and, in the case of a boiling sugar solution is dependent not upon density only but also upon the purity of the liquor and the temperature at which evaporation may be going on at the moment the critical density is reached. When then, this condition has been attained in the estimation of the operator, "pan-man" or "sugar boiler," he must so regulate matters that at this stage the control of the eventual product shall be completely at his command. Obviously, quantitatively, at least, conditions are fixed for him; no more than a definite quantity of liquor, at a uniform density can be taken into the pan, and this liquor contains a determinable weight of sugar, so that it follows, if a predetermined size of crystal be required, he must limit the number of crystals born with due consideration of the size they are to attain. If a large crystal be wanted he must see to it that he limits the birth of the crystals so that he shall have sufficient sugar in the liquor to allow them to grow to the dimensions expected.

At this stage, then, close attention is necessary in order that at the proper moment crystallization shall be checked, or rather, that the formation of an undue number of crystals shall be prevented. As the critical moment approaches, the pan-boiler takes samples of the boiling liquor out of the pan by means of a proof-stick. This is a metal rod about 1 inch in diameter and about 2.5 feet long, freely running in a sleeve in the side of the pan. The sleeve has a ground face against which a ground shoulder on the proof-stick fits in such a manner that fairly tight contact is made and air is prevented from leaking into the pan. Near the extreme end of the stick there is a cup-like depression in which about an ounce of liquid can be drawn from the pan. As the pan-man draws the sample out, he throws the liquid on to a glass plate, or allows it to flow between his finger and thumb, at the same time examining it before a bright light. If the density of crystallization has been reached, the first young crystals will appear as tiny points of light flowing down the glass, and it will be necessary to watch the rate of increase very closely. The moment the operator judges that enough of these crystals have been formed, he quickly opens the valve communicating with ST, from which a supply of liquor may be drawn into V. The inflowing thinner liquor brings the boiling mass to a density at which no more crystals can be formed, but those already formed, begin to grow as the density of the liquor again begins to rise. It is now that the nursing of the crystals goes on, and in order that they shall grow in vigor and strength, they are treated to occasional "drinks," *i. e.*, liquor is drawn in whenever the density of the mother-liquor (syrup surrounding the crystals) tends to rise to the point at which a new lot of crystals would be born; if this last should happen trouble would result, for the second crop, coming into the world so much later than the first, would not grow to the same size as their older brethren, and thus the resulting sugar would show an uneven grain when finished and the washing in the centrifugals would be difficult. When the boiling mass, which is now a mixture of crystals floating in the mother-liquor, through repeated drinks, fills the pan, it is finally brought to a proper consistency and the boiling is finished.

Control of temperature and rate of evaporation is obtained by judicious manipulation of the condenser water and the amount of steam, live or exhaust, admitted to the pan at any particular time.

When the grain in the pan has been fully developed and has attained the dimensions required, the "strike" is about ready to be discharged. If the operation has been properly carried out, the material in the pan should be at about the level of the broken line in V, and this level should be reached coincident with full grain development. Hence no more liquor is taken into the pan, but concentration is continued until the mass contains about 10 per cent water, which means that the syrup surrounding the crystals will contain from 15 to 17 per cent water.

As soon, then, as the proper density has been reached, the steam is turned off; air is admitted to V and C by opening a valve properly placed at C; AP is stopped and condenser water also turned off. Valve F may now be cautiously opened and the contents of V discharged into M. This mixture of sugar grains and syrup is known as "Massecuite" (boiled mass), "Fillmass" or "Fullmasse," and is kept in constant motion by slowly revolving paddles in M, so that the heavy grain shall not settle to the bottom of the vessel and there solidify as would be the case if it were allowed to remain quiescent. Its temperature should not be more than 150° to 155° F.

Under normal conditions, with pan and air-pump in good repair, and an adequate supply of steam, the period of boiling should be between 1½ to 2½ hours according to the density of the liquor and the size of grain required.

Centrifuging or "Spinning."—The massecuite in M is now spun in the centrifugals as expeditiously as possible, to avoid an undue quantity of "false grain" from being formed. This false or secondary grain formation is due to two causes, the partial cooling of the massecuite and the high concentration and purity of the syrup around the grain. The syrup, in the first place, is "supersaturated," *i. e.*, it contains more sugar in solution than it can normally hold for any length of time at the temperature at which the strike was discharged. Add to this the cooling in M as a further contribution to the crystallizing tendency, and we have an unavoidable factor of secondary grain formation; but rapid spinning is the only remedy to reduce the phenomenon to a minimum.

Although the liquors from which the massecuite has been boiled may have been but slightly colored, the product now in M may have a distinct deep yellow or brownish yellow color, due, of course, in the first place to the fact that the volume of the massecuite is about half that of the original liquor used, and, secondly, a certain amount of color has been formed during the boiling. The method of spinning will therefore be varied somewhat according to the depth of color and the nature and size of the grain. Deeply-colored massecuites will require that smaller charges be drawn from M into the centrifugals in order that the dark syrup in such a case may be completely washed away from the crystals.

A single centrifugal operation may be described thus: A quantity of massecuite is allowed to enter the centrifugal through a spout in the bottom of M, and the flow cut off when the operator judges that the quantity drawn will yield a wall of sugar, say, 4 to 5 inches thick after the centrifugal force of the revolving machine will have forced the charge up and against the wall of the screen. As the machine gains speed the syrup is driven through the screen and time is allowed to make this as thorough as possible. The outflowing syrup runs into one of the twin troughs, g. As soon as no appreciable amount of syrup is being purged from the sugar, a quantity of clean, filtered water (from 2 to 4 gallons per charge) is sprinkled, either from a can or by an automatic device, against the wall of the now nearly white, syrup-free crystals in the centrifugal. The water being violently forced through the crystal mass carries with it, by mechanical attrition and solution, the last traces of any adhering syrup. In the meanwhile the direction of the spout has been changed so that this wash-water, "washings," may be discharged into the other member of the twin trough.

Finally the machine is allowed to run until the water has been forced away from the crystals to such an extent that the water content of the mass shall not exceed 2 per cent. The moist crystals are then discharged through a valve-opening in the bottom of the centrifugal, dropping into a screw conveyor (con.) and by a suitable elevator carried into gB for subsequent drying, termed "granulating."

The object of separating the washings from the green syrup is but carrying out one of the guiding principles in refining operations, *viz.*, the separation of high-purity from low-purity solutions whenever opportunity offers. Varying in accordance with the original purity of the liquors which entered the pan, the syrups flowing from the centrifugals may fluctuate from 82 to 92 purity, but the washings will invariably be from 5 to 6 degrees higher, so that if a 90 purity syrup should be the product from a certain massecuite, 95 to 96 purity washings would be obtained. Such washings, brought to the proper density would be pumped to one of the syrup tanks and in due course form one of the boiling charges for a subsequent strike.

The green syrup, however, according to its purity and color, will be either sent back to the char-house for refiltration or used for boiling softs or possibly for "remelts" (q. v.).

Granulation.—Granulation, in principle, is simplicity itself, but there are a few factors to be observed that apart from the drying of the grain, contribute to the final attractiveness of the sugar.

The drying should be done with an ample supply of air, cool and dry as possible, and drawn into the revolving granulator by means of a powerful fan. The necessary heat supplied by the steam drum in the axial centre of the granulator, or from a bank of coils should be regulated by an automatic steam valve and set to dry the sugar and no more, delivering it at the mouth of the granulator as cool as is consistent with the requirements, *viz.*, the outpouring crystals of sugar should not contain more than 0.10 per cent of water and should not be more than 90° to 100° F. when filled into bags or barrels.

The most objectionable result of "over-drying," or a too rapid drying is observed in the lustreless appearance of the grain *en mass*, giving the sugar a dull, dead whiteness instead of a living sparkle. Lack of lustre may be due also to badly-formed crystals, or rather to the presence of an undue amount of "false grain." But the distinctive dullness originating in the granulator can be readily detected by examining a few grains under a low power of the microscope. In the latter case the large, normal grains will be seen to have their faces, and even their edges, covered with minute but densely crowded grains, that have been formed on the grain while passing through the granulator. On the other hand, the comparatively larger false grain, adhering to the large normal ones, may be said to have been baked upon them during the drying. This distinction between these two conditions will enable the observer to at once determine to which of the two causes the dullness may be due.

With the observance of these few essential conditions granulation proceeds satisfactorily provided the mechanical conditions be right.

SOFT SUGAR PRODUCTION

As compared with granulated, the production of an attractive and profitable soft sugar involves much more detailed and varied supervision. From the time the liquors and syrups, from which the soft is to be boiled, have been selected and up to the final disposition of the finished product, there intervene a series of interdependent operations that each in their turn may influence the final result in a marked manner. The varied grades of softs usually called for, demand that the liquors destined for each grade shall be of the proper purity, density and color, and that the massecuite shall have been discharged from the pan under quite narrowly-defined conditions in order that this massecuite may yield the sugar it has been sought to produce. Again, the composition of the soft sugars of equal commercial value may vary to a considerable degree, and in such a manner that one particular lot may be more attractive or more profitable, or both, than another commanding the same price. In the face of these facts, a detailed discussion of the requisite conditions for successful soft sugar production is obviously necessary.

Selection and Preparation of Liquors and Syrups.—Public demand and industrial requirements are such that a wide range of soft sugars may from time to time be called for, and in order that a particular strike shall yield a massecuite from which the required sugar can be spun, the composition of the liquors entering the pan must be determined to a nicety. In this respect no particular instructions can be laid down as guides for producing intermediate grades, suffice it to say, however, that under normal char-filtration a liquor of from 91 to 93 purity should yield a soft sugar of the highest quality in color and texture. Relative to lower, but still superior grades, selection of the liquors must be left to the experience of those usually charged with the supervision of the operations directly connected with this line of work. As regards the general conditions, however, of liquors and syrups entering into the composition of the material to be boiled definite qualities can be profitably indicated.

Color and Brilliancy.—Both liquors and syrups in respect to color should be of distinct yellow tint rather than brown or reddish, but even this may be tolerated provided that the solutions be brilliant, *i. e.,* not clear only when viewed by transmitted light, but also free from opalescence when observed by reflected light. (See Tintometry).

Purity and Density.—The purity of a liquor is only remotely related to color, as the latter is subject to great fluctuations coincident with all the possible variations of refining operations and the nature of the raw material entering the house. Indeed, under favorable conditions a comparatively low-purity liquor may carry little color and in such a case furnish profitable material for the production of a high-grade soft sugar. In short, a wide range of purity may be allowed in the manufacture of high and medium-grade softs so long as the color of the liquor is sufficiently attractive to ensure the final product. As a matter of fact, a low purity is to be preferred as will presently be explained.

The density of the liquor, however, is a matter that should be given attention as the eventual polarization of the finished sugar is in a large measure influenced by this factor. Liquors and syrups entering the vacuum pan should not be under 57° Brix at normal temperature, as otherwise the pan-boiler cannot readily produce a grain that will contribute its best qualities to the sugar.

Reaction and Sulphuring.—On general principles, of course, liquors should never exhibit a distinctly alkaline reaction when issuing from the char-filters, and when this is the case, correction should be made upon the raw liquor rather than upon the filtered liquor. But if filtrates destined for soft-sugar production should exhibit alkalinity this can usually be corrected by a more generous sulphuring of the liquor before it enters the pan. But even a normally acid liquor is with advantage "sulphured" to a higher acidity immediately before being drawn into the pan.

Sulphuring is accomplished in several ways. The fumes of burning sulphur are either bubbled through a column of the liquid, absorbed by a stream of liquor flowing against a current of the sulphurous gas, or, lastly, the gas is absorbed by water and a quantity of this sulphured water added to the liquor—a method now seldom, if ever, used. Adsorption by counter-currents of gas and liquid is at present almost exclusively practiced. Sulphur is burned in a specially devised oven and the resultant gas (SO_2) pumped or injected into the absorbing tub.

The gas is rapidly absorbed by the liqour passing through the tub, and has a decided "acid reaction" when the operation has been completed. This acidity of the liquor should be ample but in no manner excessive. Apart from the implied waste of the superfluous sulphur, the presence of too much free sulphurous acid may result in the formation of free sulphuric acid and sulphates, both decidedly objectionable constituents of soft sugars. It is therefore well to establish a prescribed method of sulphuring which shall fairly hold the SO_2 contents of the liquor within set limits, but which may be varied from time to time if any fluctuation in the quality of the liquors be observed. Once a sulphuring procedure has been fixed, based on a fairly uniform consumption of sulphur and rate of flow, in time and quantity, of the liquor passing through the apparatus, tests determining the quantity of sulphur absorbed need not be made upon every batch of liquor treated. It is well, nevertheless, to have SO_2 determinations made at regular intervals as a check upon the operator as well as to be assured of the steady efficiency of the sulphuring apparatus.

A sulphur figure may be represented in any convenient manner, but perhaps the readiest method of presenting the SO_2 contents of a liquor may be stated in milligrams of SO_2 in a liter of liquor. In practice this figure should not be below 200 and certainly never above 400 milligrams; 250 to 300 will usually be ample. (See reference, Control).

Composition of Material for the Vacuum Pan.—The liquors and syrups, having been selected and sulphured as indicated, are mixed in such proportions that the final purity and color of the resulting massecuite shall yield the sugar required.

The liquors to enter the pan are, of course, the char-filtrates gathered for this purpose as shown under Separation of Char Liquors, page 77. The syrups may be those held in reserve from previous boilings of either granulated or soft sugars, and may range in purity from 82 to 92 from granulated boilings, and from 50 to 80 in the case of softs. The granulated syrup may be used with "A" or other liquors, while those from the softs will be used to reduce the purity of the liquors and granulated syrups, if these are too high for the purpose required. Furthermore, as will presently appear, these lower grade syrups are also useful for controlling the purity of the massecuite and for special final charges to the vacuum pan when certain low-grade softs are to be made.

But beside the purity control in this respect, other conditions must be observed and maintained to assure the very special features each grade of soft sugar should possess to make it profitable as well as attractive. Not only will the boiling be facilitated, but centrifuging cost will be held to a minimum if the pan-man receives the material under conditions that will enable him to grow and build up a proper grain and deliver a massecuite of desirable properties. Especially is this necessary when a low-grade sugar, polarizing from 83 to 84 is to be made. In such an event a final massecuite purity of not more than 73 or 74 must be reached.

THEORY AND PRACTICE OF SOFT SUGAR BOILING

It is a truism regarding the phenomenon of crystallization that the purer the solution in which crystallization takes place the more rapidly do the crystals form and the more truly do they conform to the system to which they belong. In practice this is more particularly marked of those solids from which viscous solutions are obtained, and the viscosity, in the case of a sugar solution, rises very rapidly as the purity falls; hence it follows that high-purity liquors crystallize much more rapidly than those of lower purity. Indeed, a point of low purity can be reached at which only special and prolonged effort can induce crystallization at all. But as far as this law affects the practical features of soft sugar boiling, certain general data have long since been established, and their successful application is a matter modified somewhat by conditions of prevailing methods and the knowledge of the liquor composition other than indicated by the purity. It follows, then, that if difficulties are to be encountered, they are likely to be accentuated when low-purity material is to be crystallized. Therefore the factor essential to the proper boiling of low-grade sugars should be studied with care and applied in every possible detail.

Evidently then, a discussion leading up to such factors, and a general review of the component liquors for the vacuum pan are in order here.

Having determined the cubic capacity of any particular pan, it is an easy matter to estimate the weight of massecuite a normally filled pan will deliver, and from such a figure to arrive at the volume of liquor at standard density, say 54.5° Brix, that the apparatus will be able to receive.

In the case of soft sugar-boiling the entire material taken into the pan may be divided into three parts, or charges, each having a particular function to perform towards the completion of the operation.

Vacuum Pan Charges.—Such charges may conveniently be designated as:

I. Boiling-down Charge.
II. Graining Charge.
III. Final Charge.

Within slight variation these charges may be respectively four, three and two parts of the total solid material, in liquors and syrups, to be taken into the pan; in the case of better grade sugars, Charge III may be omitted altogether, Charges I and II would then be four-sevenths and three-sevenths, or even five-sevenths and two-sevenths respectively.

Charge I is usually all drawn into the pan at once and evaporated down until grain begins to show, and then Charge II is drawn in in quantities determined by the rate of evaporation and at the moment the pan-man may require liquor for the development of the grain. If the boiling has been properly conducted and the composition of the liquors has been correctly judged and calculated, this graining charge should suffice for the perfect development of the grain by the time the charge has been exhausted and the now fairly heavy massecuite will be ready for the final charge. This, in the case of low-grade sugars, should be a heavy syrup (70° to 75° Brix) of low purity, between 50 and 60, and be drawn into the pan as rapidly as the massecuite will absorb it. The reason for its introduction will appear presently. (See page 95).

Composition and Function of Charges.—The function of Charge I is to supply the material in which and from which the first grain shall be born and also to serve as a source of sucrose supply for the development of the young grain. As already indicated on page 80, the grain born at the time that the critical density has been reached, must be nursed to the required final dimensions by the judicious drawing in of charges of light liquor, thus keeping the boiling liquid at a density just sufficient for the growth of the crystals without permitting a new crop from being formed. This is, then, the primal function of Charge II. But in soft-sugar boiling a second crop of crystals is always desirable for a very specific purpose. The requisite quantity of this second crop of grain must be carefully introduced at the proper time so that it may be sufficiently developed when crystallization has been completed.

The massecuite, before the introduction of the third charge, should contain, therefore, two distinct sets of crystals, *viz.*, those first formed and which will be larger, somewhat harder than the second lot, which will be small but more numerous than the first lot. This second group of crystals is proportioned to just fill the interstices between the larger crystals when the finished massecuite is being spun in the centrifugals. The proper proportioning of the second group, as to size and quantity, is one of the most important operations of the entire boiling. The successful production of sugar from the massecuite is very largely dependent upon the judgment of the pan-man in this respect. If the second grain should be either too small or too numerous, or both, it will pack during the centrifuging and thus prevent the syrup from being properly purged from the sugar crystals. Indeed, so much syrup may be held by such a badly proportioned crystalline mass that enough syrup will be persistently retained by the sugar to make it soggy or lumpy, or in extreme cases render the sugar entirely unworkable, resulting in the necessity of a remelt.

Purity of Charges.—To facilitate the efforts of the sugar-boiler in the production of this all-important grain mixture in the pan, a very definite purity of the charges has to be determined. This so-determined purity falls back upon the facts given under Theory and Practice of Soft Sugar Boiling, page 88. In this respect, practice rather than theory has fixed the purity figure for low or medium-grade softs. This first charge should for these have a purity between 82 and 84; when high-grade sugars are to be made, the purity is usually higher than this and the composition is further influenced by color considerations.

However, it is frequently advisable when bright or medium bright sugars are to be made that the first charge for such a boiling be given at about 86 to 88 purity and the second charge of higher purity. For instance should it be known that a massecuite purity of say 90, will yield a required grade of soft sugar, a first charge of 88 should be followed by a second charge of 92, making a final purity of about 90. This procedure is to be preferred over a uniform purity of 90. Two charges of 86 and 94 might even be used if liquors at these purities are available.

The reason for adopting such a procedure is to insure the production of a "soft grain," and it is known that purities in the neighborhood of 86 are most appropriate.

As regards low-grade softs, the second charge may with advantage be as low as 70, rising to 78 or 80 at times. It is evident, therefore, that these two charges should be carefully calculated as to purity and quantity, and as the liquors and syrups available are not usually on hand at the required purity, mixtures must be made of high and low-purity material in process for the purpose. The charges will be drawn from the "A" liquors and the syrups stored from the boilings of various grades.

Calculating Purity of Charges.—In practice those charged with the duty of ordering the mixtures for the pan must determine the quantity of each constituent of a charge that shall be of the calculated purity and compounded from materials of known composition.

A convenient formula is necessary so that the calculation may be quickly and accurately made. The ultimate arithmetical work is quite simple and is based on the following equations, when but two liquors or syrups are concerned—the introduction of a third liquor is quite unusual and need not be considered here.

If

M = Cubic feet of mixture required.
P = Required purity of mixture.
a = Test of liquor of higher-purity material.
b = Test of syrup or lower-purity material.
x = Cubic feet of "a" required.
y = Cubic feet of "b" required.

Then,

$$x + y = M$$
$$ax + by = MP.$$

By elimination we get:

$$ax - bx = MP - Mb$$
$$x(a - b) = M(P - b)$$
$$x = \frac{M(P - b)}{a - b}.$$

This simply means that the difference of P and b, multiplied by the cubic feet of the mixture required, is divided by the difference of a and b. Then, of course,

$$y = M - x.$$

Example.—How much liquor at 90 purity and how much syrup at 60 purity will be required to make 400 cubic feet at 84 purity?

$$x = \frac{M(P - b)}{a - b}.$$

By numerical substitution

$$x = \frac{400(84 - 60)}{90 - 60} = \frac{9{,}600}{30} = 320$$

$$400 - 320 = 80.$$

Proof

$$\begin{array}{rr} 320 \times 90 = & 28{,}800 \\ 80 \times 60 = & 4{,}800 \\ \overline{400} & \overline{33{,}600} \end{array}$$

$$\frac{33{,}600}{400} = 84^\circ \text{ purity sought.}$$

Cobenzel's Diagram.—Calculations of this kind may also very conveniently be made by the aid of Cobenzel's diagram, but the results will be obtained in "parts," whereas by the preceding method the actual volume required is directly obtained.

This is the method:

Draw a rectangle and join the corners by diagonal lines. At the left-hand corners of the rectangle note the purities of the liquor and syrup to be used, and at the intersection of the diagonals note the purity of the mixture desired. Find the difference between this number and the two given numbers and note the results at the opposite right-hand corners at the ends of the corresponding diagonals. The results thus obtained show the relative number of parts required of the given liquor and syrup which lie on the corresponding *horizontal* lines of the rectangle.

To illustrate:

We learn that twenty-four parts of liquor (purity 90) and six parts of syrup (purity 60) will result in a mixture having a purity of 84.

But as 400 cubic feet are required, we can obtaian the volume of liquor thus

$$30:24::400:x = 320.$$

The volume of syrup required will be:

$$400 - 320 = 80 \text{ cubic feet.}$$

Hence the mixture of 400 cubic feet at 84 purity will be obtained by mixing 320 cubic feet of liquor with 80 cubic feet of syrup.

These calculations are based on the assumption that both liquor and syrup are at standard density, *i. e.*, 54.75° Brix. But either of the liquids may be heavier or lighter than the standard or normal Brix. Corrections will therefore have to be made on the number of cubic feet found by the formula, not only to adjust their proper relative proportions, but also to establish a volume equal in solid contents to the recognized or adopted density standard. For this purpose a convenient and sufficiently accurate procedure may be, to increase or decrease the volume found by one-fortieth for every degree Brix above or under standard.

Thus, suppose in the example cited, the liquor to be 52.5° and the syrup 60.5° Brix respectively, the corrected volume then would be found as follows:

$$\frac{320 \times 2}{40} = 16;\ 320 + 16 = 336 \text{ cubic feet corrected volume.}$$

$$\frac{80 \times 6}{40} = 12;\quad 80 - 12 = 68 \text{ cubic feet corrected volume.}$$

But if a more accurate adjustment be required the Table of Factors given below may be employed.

TABLE VIII.—TABLE OF VOLUME CONVERSION FACTORS

Deg. Br.	Lbs. solids per cu. ft.	Factor	Deg. Br.	Lbs. solids per cu. ft.	Factor
45	33.83	1.27	59	47.25	0.91
46	34.73	1.24	60	48.26	0.89
47	35.64	1.20	61	49.29	0.87
48	36.56	1.17	62	50.33	0.85
49	37.49	1.15	63	51.38	0.83
50	38.44	1.12	64	52.43	0.82
51	39.38	1.09	65	53.50	0.80
52	40.33	1.06	66	54.57	0.79
53	41.29	1.04	67	55.65	0.77
54.75	43.00	1.00	68	56.73	0.76
56	44.22	0.97	69	57.84	0.74
57	45.21	0.95	70	58.96	0.73
58	46.23	0.93			

For example:

$$80 \times 0.88 = 70.4$$
$$320 \times 1.05 = 336.$$

Of course, temperature corrections are not necessary since in practice the error due to small temperature changes may be neglected.

CENTRIFUGING SOFT-SUGAR MASSECUITE

If all the conditions have been observed and the preparations carried out as described relative to liquors and syrups destined for a soft sugar, the pan-man should be able to produce a massecuite from which the required sugar can be spun.

During the boiling for soft sugar the temperature of the boiling mass should be kept as low as possible, should never rise above 145° F., even when the first grain is being formed and should generally be in the neighborhood of, say, 130° F. Towards the end of the boiling and when the pan is nearly full and the massecuite has reached its discharging density, a rise of 5° to 8° is no longer objectionable. When high-grade softs are to be made the pan operations are completed immediately after the second charge has been drawn into the pan and the massecuite concentrated to a water content of from 7 to 8 per cent, or to a consistency of a fairly free-flowing mass. The massecuite is now ready to be discharged into the mixer.

If, however, a medium or low-grade sugar be sought, a final charge of a heavy, low purity syrup, reserved from a previous boiling, is drawn into the pan immediately after the boiling has been completed in other respects.

Final Charge.—The purpose of this final charge is to act as a diluent of the heavy, tenaceous massecuite usually the result of boiling low-purity material used for this class of products. Indeed one of the most important aims in the production of these soft sugars is to secure as low a polarization as is consistent with the nature of the finished sugar. This is in a large measure determined by the proper proportioning of the first and second grain, and facilitates the centrifugal work, as already indicated. But a thin, too freely purged massecuite would not readily admit of the retention of the requisite amount of syrup, while a stiff massecuite would act in the opposite direction. Hence, the massecuite is boiled to a very low-water content and the final charge added to restore the desired and necessary fluidity. However, the full significance of the final charge in this respect will become more apparent in the paragraphs dealing with the crystallization of the lowest products of a refinery, *viz.*, the production of black strap and syrups destined for barrel syrups.

Centrifugal Work.—The centrifugal work on soft sugars generally, and the low grades particularly, requires considerable attention and skill. The advantages obtainable from a normal massecuite may be entirely lost if this work is not carried out with judgment and care. The composition of the finished sugar can be largely controlled by proper operation at this station. It is well known that a certain percentage of the syrup, in the form of an adhering film, should be left in the sugar as it is discharged from the centrifugal. And as the syrup carries, of course, nearly all the non-sucrose constituents, the sugar will contain these in direct ratio to the amount of syrup it may be able to hold, which in its turn is determined by the nature of the grain and the centrifugal work.

Assuming an adequate grain structure, the question remains; how may centrifugal work make or mar the good qualities of a sugar? Obviously, by purging out too much or too little of the necessary syrup. But in order to control the amount of this syrup, the quantity of massecuite to be taken at one machine charge has to be nicely judged and the time for spinning regulated to meet the demand. While similar grades of softs admit of general working methods, there are nevertheless, it may be said, idiosyncracies to be observed in every strike, not infrequently attributable to the "personal equation" of the panman himself.

Following the cycle of a machine from charge to discharge, it will be noted that as the syrup begins to leave the crystals, the true color of the sugar will shortly become apparent on the inner and visible surface of the spinning mass. But the ring of sugar lying against the screen of the machine will always be found to carry more syrup than the inner ring. Indeed, the depth and quality of this outer syrup-carrying ring will eventually in a measure determine the quality and nature of the finished sugar. If this syrup ring in the machine be thin and sharply defined, it is always accompanied by difficult machine work, and unless extraordinary care be taken the resulting sugar tends to be lumpy. If, on the other hand, the ring shades into the dryer part of the sugar wall in the machine, it is an indication that the pan-man has produced a desirable massecuite; centrifugal work will run smoothly and the sugar will be free, without lumps, of woolly texture and carry the maximum amount of syrup constituents with other desirable properties.

Modern self-discharging machines are unsuited to soft-sugar work. But the best centrifugal work on soft-sugar massecuite can never yield a uniform sugar as it leaves the machines. The freshly-spun sugar must be "homogenized," *i. e.*, the more syrupy portion, which comes from the screen, and the dryer portion of the inner part must be thoroughly incorporated one with the other. The screen sugar carries too much syrup; the inner sugar too little, and the two must therefore be properly mixed to produce the normal sugar.

This mixing is best achieved by passing the sugar as it comes from the machines through a system of rollers and then through a "brush," as suggested in the sketch.

Allowing the sugar to pass through a "cooler," placed between a roller at the end of con. (not shown in Diagram VI), the roller and the brush, has a further advantage in this, that the sugar will be cooler upon packing.

The "cooler" is a revolving cylinder, in fact a short "Granulator" without the heating appliance, while the fan at the rear of the cooler draws cool air through the cylinder.

Composition and General Character of Soft Sugars.—No other products of the refinery are subject to such divergence in physical appearance and chemical composition as those sugars generally designated "softs." Often operations quite remote from the final stages of soft-sugar production have a pronounced influence upon the character and commercial value of the finished product.

Indeed, certain raw sugars will directly determine some properties of the softs made from them, nor is it always possible to entirely eliminate all undesirable effects of the raw sugar from making their appearance felt in the refined softs. At all times, then, any attention paid to the factors involving soft-sugar production will be found to benefit this so critically examined product.

As soft sugars find a wide use in the preparation of foods and sweets, many different grades are frequently called for, and so the refiner catering to soft-sugar supply may be required to make shades of this product ranging from a dark yellow or brownish tint to an almost white or cream-colored sugar. This means that such a range of sugars must vary commensurately in chemical composition, and that their grain structure and general texture must likewise differ in many ways. Setting side by side the extreme analyses of soft sugars, a long list of intermediate compositions can be imagined.

Softs, then, may range in analysis from:

	Dark	Bright
Sucrose	83.0	95.0
Invert sugar	6.0	0.8
Ash	2.0	0.3
Organic non-sugar	5.0	0.9
Water	5.0	3.0

Generally speaking, sugars testing 90 to 95 should not be made to contain more than 3 or 4 per cent of water, while intermediate grades can with advantage be made to carry 4.5 or even 4.75 per cent water, more especially if the sugar in question has been well boiled and exhibits a "woolly" grain.

The control of the other constituents is, of course, more remote, but given the required char-filtered liquors, especially if these be bright and carry an adequate amount of invert sugar a very attractive soft of any grade can be made to polarize moderately and retain a goodly amount of syrup, which, in its turn will be the medium of introducing a desired amount of invert sugar that contributes so much to the keeping qualities of the sugar, since the syrup contains nearly all the invert sugar and the non-sugar. A soft with an adequate amount of invert sugar will not readily harden and dry out during storage—nor can the deficiency of the syrup retaining power of a sugar be made up by moistening with water. The addition of water dissolves some sucrose, raises the purity of the contained syrup with a strong tendency toward recrystallization of this sucrose consequent upon a slight evaporation of water while in store. It is no doubt the comparatively high ratio of invert sugar to sucrose in some softs that inhances their keeping qualities.

Finally, then, a desirable soft should have a woolly grain structure, be rich in invert sugar and low in ash, and hold enough syrup to enable the sugar to contain 4 to 4.5 per cent water. Such qualities can be most readily imparted to a soft when no black strap and little refined syrup is being made, since black strap and syrup production throws much of the invert sugar into these products with the result of low invert in the soft sugars.

FANCY SUGARS

Under this term may be gathered the lengthy list of concreted sugar crystals in the form of cubes, tablets and parallelograms. While the mechanical devices for the formation, handling and packing of such sugars are almost as numerous as the forms and sizes produced, the basal methods used for plate and cube production are limited to two distinct procedures. One may be considered essentially mechanical. the other rather chemical in principle. This last—which will not be discussed in detail—depends chiefly upon a secondary crystallization induced among a mass of sugar crystals forced into molds of the required form and size. The so-formed cakes are dried and finally sawn to dimensions desired. The several processes based on this principle, while yielding very attractive products, are expensive, involving much machinery and are wasteful in that they produce much "scrap" to be returned for recrystallization.

Adant and Scheibler Processes.—This concrete crystallization as applied to cube sugar work may be regarded as an evolved method of the old sugar-loaf production. Indeed, much of the cube sugar produced in Europe to-day is sawn from the sugar-loaf.

The Adant and Scheibler Methods differ only in their mechanical applications, both depend upon the rapid concrete crystallization of a massecuite of high purity.

Mechanical Processes.—The more distinctly mechanical processes, depending almost entirely upon pressure for the formation of the cube or plate, are much simpler, less costly and certainly reduce "scrap" to a minimum. With due precaution the products obtained by mechanical molding can be made to rival their more expensively produced competitors. Excellent results can be obtained by an extra filtration of a high-purity liquor over superior bone-black and from the resulting filtrate boiling a massecuite of a definite but distinctly mixed grain. At least three grainings should be made: the first batch of grain formed should be limited to about 20 per cent of the entire boiling and should be well on the road to development before the second, and much more numerous group is permitted to appear. Ample time should be given to this second group for development and growth. Lastly, about 20 to 30 minutes before "striking" a third grain is induced as rapidly as possible, and as the time for growth of this last and third lot of crystals is very limited this grain group will be very small at time of striking. If the three groups of crystals have been judiciously developed as regards to relative quantity and size of grain, the pan will discharge a massecuite from which may be spun a sparkling, moist white sugar available for either light or heavy pressing in the molding machines.

The centrifuging should, of course, be carefully done; large machine charges should be avoided as the mixed grain tends to retain the syrup and it is advisable to use about 30 per cent more wash water than for ordinary granulated sugar work. The spun sugar is stored in any convenient bin and conveyed to the presses with an addition of such a quantity of white liquor (saturated sugar solution) that the plastic mass entering the presses shall contain from 1.5 1.75 per cent of water. When particularly hard cubes are to be made and a comparatively high pressure is to be used, the lower percentage of water must be maintained as otherwise the resulting cubes will be a dull white and the sparkle of the larger grains entirely lost.

The importance of securing a mixed grain during the boiling becomes very apparent in the finished product. The large grain gives tone and sparkle to the sugar, while the smaller grain particularly contributes strength and weight to the cubes and facilitates press work. In fact a deficiency of this small grain may require correction through the addition of a quantity of powdered sugar introduced at the time when mixture is made with the white liquor. But the so-added powdered sugar is not in any way as efficient as the small grain and never gives the resulting cube the life and sparkle desirable.

Press Work.—The compression that the plastic mass may require or endure without destruction of the enlivening effect of the largest crystals upon the surface of the cubes, depends largely upon the duly-proportioned mixed grain in the boiled mass.

In the case of pressing individual, comparatively thin tablets the limits permissible are to be determined by one-thirty-second of an inch; two-thirty-seconds, or at the utmost three-thirty-seconds, other conditions being right, will ensure a sufficiently hard tablet, readily soluble in use.

Bars and plates of a finished thickness of 1 inch may be compressed three-eighths of an inch, and if the grain is favorably proportioned the filled mold, one and seven-sixteenths inches deep, may be compressed to 1 inch. Cubes cut or sawn from such strongly-pressed bars or plates will be extremely hard and resemble very closely the products from the Adant and the Scheibler Methods.

Drying.—Where ample space is available, the more lightly pressed tablets can be dried without any artificial heat; 24 hours will usually suffice. Drying in a current of warm air is, however, to be preferred, as the rapid drying, in from 3 to 5 hours, depending on temperature and the volume of air passed over the product, no doubt gives firmness and a more pronounced glitter to the tablets. The temperature of the ovens should never exceed 145° F., and at this temperature an ample, dust-free air should flow steadily over the drying cubes. If time and oven space permit, it is better to dry at 130° to 135° F.

Bars and plates always require heat drying, and while the heat of the ovens may be kept, in case of necessity, at the maximum indicated for tablets, lower temperatures are always advisable, since the period of drying runs from 20 to 24 hours and long exposure to excessive heat will develop distinctly yellowish tints in the bars.

Although the oven system may be very simple in principle any design of a system should be carefully calculated to deliver sufficient air, at a determined degree to effect the drying in a specified time in order that the slightest coloring of the product be avoided.

CHAPTER IV

INTERMEDIATE AND END-PRODUCTS

INTRODUCTORY

It will have been noted that in most of the operations described in the preceding three chapters that some residual material had accumulated after the main products of the process had been obtained. Generally the intermediate or residual substances, usually syrups and sweet-waters of some kind, were not dealt with at all but deferred to subsequent pages—in short, the direct refining operations were followed along the lines of chief production only. It is therefore now necessary to take up the disposal of these intermediate products and to show how they must be brought, by appropriate means, to contribute to the general operations of the house, or yield the sugar they carry to the sum total of the refined products.

Indeed, the economic disposal and adsorption of the varied intermediate products is one of the besetting problems which the superintendent forces are constantly called upon to regulate and guide. As it is one of the main endeavors of the refiner to keep all sugar solutions of similar purity from mingling with higher or lower ones, the disposal of the intermediate products must be regulated to meet this desirable condition as thoroughly as possible. Frequently, also, the color and physical condition of the material has to be considered as well. From time to time the method of handling some such product has to be changed to accommodate altered conditions in the refining operations or to meet any exigency of the house in general, or as imposed by a call for special products. Usually a predetermined method is followed as strictly as possible and the sweet-waters and syrups of all kinds flow along well established routes in the operations of the house.

SWEET-WATERS

The most troublesome and the most costly of disposal are the sweet-waters, an embrasive term for all thin sugar solutions, say, from 50° down to 3° Brix, that must be concentrated to a work-able density if they are not of a composition to serve as diluents of heavy syrups or liquors. Volumes of sweet-water are accumulated from many sources.

The main and characteristic origin of sweet-waters are from (1) the char-filters, (2) filter-presses, (3) bag-filters and (4) raw sugar bag washing tubs. Steamings from pans, tanks and machinery; floor washings, etc., are collectively of small volume and need no particular mention as they are easily disposed of and are run to one of the several tanks receiving sweet-waters. The other four sources of sweet-water require some discussion.

I. **Char-filter Sweet-water.**—The sweet-water from the char-filters are and should be fairly constant in quantity, purity and density, more particularly when averaged over a day's run. As the number of char-filters in daily use does not vary, or very slightly, and each filter, or battery of filters receives in the same given time the same amount of sweetening-off water, following upon liquors, which in their turn are similar in purity, the average sweet-water obtained from this source is, therefore, subject to comparatively slight variation. (See page 198). A char-filter. then, as typically illustrated on page 197, would produce from 200 to 300 cubic feet of sweet-water of about 74 purity and 8° to 20° Brix, and all companion filters of the same battery would produce a sweet-water very similar in quality and volume.

II. **Filter Press Sweet-water.**—The composition of these sweet-waters varies considerably and is largely dependent upon the quality of the raw sugar being refined and the nature of the affination work. They are usually concentrated in the effects and thus mingled with the greens, or may be used directly as diluents for the greens before these are treated in the blow-ups.

III. **Bag-filter Sweet-water.**—Again referring to page 51 it will be seen that the greens (Gr.) are taken off for further treatment at 17° lower purity than the WR from which they have been purged, the greens, therefore, contain a very large percentage of the impurities of the raw sugar, in fact, as there shown, nearly 40 per cent of the O. N. S. has accumulated here. It is the presence of this O. N. S., and relatively high viscosity of the greens that has hitherto made attempts at filter-pressing them abortive, and bag-filters must be used to clear them of their suspended matter. In bag-filtration we have the reverse of press-filtration in this, that the mud is gathered within the cotton bag (page 60) while in press-filtration the mud is collected on the outer surface of the filtering sack.

But in bag-filtration, as in press work, the resulting sweet-water has a purity within the neighborhood of the syrup of its origin, and is produced by a similar method, *viz.,* when the bags of the filter become clogged with mud and in consequence the flow of the syrup stops, the mud must be sweetened-off and the bags washed.

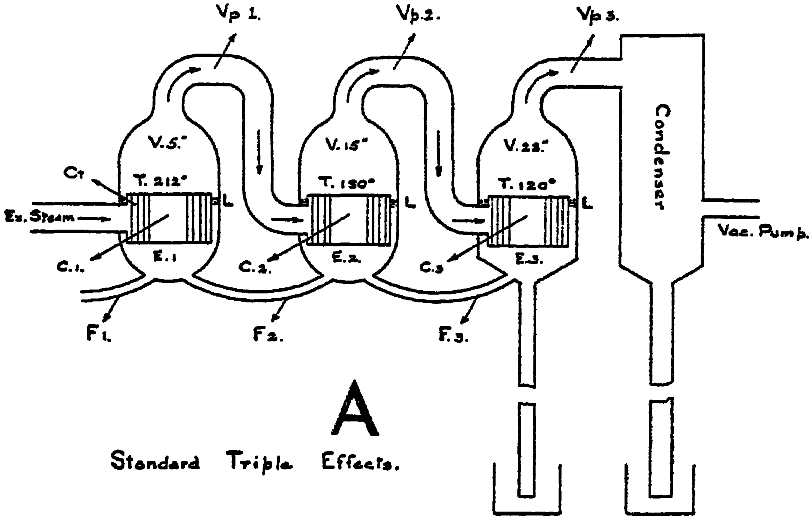
Many schemes have been tried and many methods used at one time or other to reduce sweet-water production incidental to bag-filter work. Various devices for "flushing" the bags have been found profitable, but, perhaps the simplest in this case is the best. Flushing consists of running either water or sweet-water through the bags before removing them from the filter for washing in the tubs or the washing machines. One practice is to run a certain volume of char-house sweet-water through the bags and thus reduce the sugar content in a preparatory manner. This done, the bags are taken out of the filter, brought to a series of wash tubs and washed against a stream of flowing warm water. The resulting muddy water is gathered in a mixing tub, limed to a definite alkalinity, heated and pressed as described under II. The resulting pressed sweet-water in this case is very dark but clear. As its purity lies between 75 to 85, it is either taken to the evaporators for concentration or may be used at a certain stage of remelt boiling. If this sweet-water be taken to the evaporators it is there brought to a density of 58° Brix and returned to the greens and with them reenters the refining process to which the greens are suitable.

IV. Sweet-water from Washing Empty Raw Sugar Bags.—In refineries where the removal of adhering sugar is done by washing the empty bags in hot water, or by steaming them, a sweet-water is obtained of comparatively high purity but very muddy, charged with fiber from the bags and all nondescript matter that may have accumulated upon them. For this reason and the fact that hot water washing, and especially steaming extracts considerable color from the jute, these waters are directly sent to the general sweet-water tank from which the evaporators draw their supply.

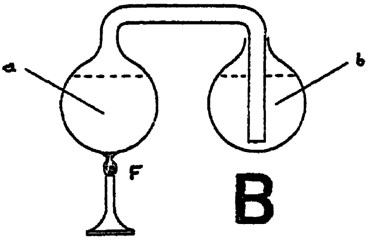
The aggregate sweet-waters of a day's operation of a refinery may be in the neighborhood of 10,000 cubic feet per million pounds of raw sugar, and as from 60 to 70 per cent of the weight of this water must be evaporated, to concentrate, say from a mean density of 20° Brix to a final density of 55° Brix, it follows that from 400,000 to 500,000 pounds of water will have to pass off as vapor from the evaporators in 24 hours. Economy both in quantity of sweet-water produced and in the method of evaporation is an apparent and important factor in this department. It is quite difficult to estimate the quantity of sweet-water that may be expected from a fixed quantity of raw sugar, as the filtering qualities of raw sugars vary widely, even within the same class of sugars. Some days the evaporators will be taxed much more than upon others and this is usually due to the large number of filter-presses and bag-filters one raw sugar may require over another. In estimating, therefore, the capacity of an evaporator installation for this purpose it is wise to leave ample margin for the handling of any adventitious sweet-water due either to a poor lot of raw sugar or some accidental disturbance in the refinery likely to throw abnormal work into this station.

Concentration of Sweet-water.—The concentration of the collected sweet-water is now universally conducted in apparatus generically known as "Multiple-Effect Evaporators" and specifically as double, triple or quadruple effects, in accordance with the number of evaporators or effects in the system. There are several types of such evaporators in use, but they all aim at steam economy by the multiplication of the effects in the system and by the uniform and active distribution and circulation of the liquid in thin layers or films over the heating surface during ebullition.

A multiple effect as especially adapted to the evaporation of sugar solutions is shown in Diagram VII. Before describing the method of operation it is desirable to consider the main features of construction.



Standard Triple Effects.



Apparatus to demonstrate the principle of Effects

Diagram VII.

The apparatus, Diagram VII, is a triple effect, consisting of three bodies, E-1, 2 and 3, each body being provided with a steam chest, called a "calandria" (C-1, 2 and 3) and it is from this arrangement that the apparatus is known as a Calandria Evaporator. The vapor pipe Vp-3 of E-3 is connected with the usual form of condenser, while the vapor pipes Vp-1, Vp-2 of E-1 and 2 are connected with the calandria C-2 and 3 of the bodies E-2 and 3 respectively. It is to be noted that the calandria C-2 and 3 have the double function of serving as condensers for the steam from the preceeding body and as the source of heat to the body in which they are located. The bodies are also connected by the flow-pipes, F-1, 2 and 3; through F-1 the thin sweet-water is drawn into E-1 and then by way of F-2 and 3 into E-2 and 3. The exhaust steam intake is the only source of external energy supplied to the three effects, *i. e.*, E-1 is the only one of the three bodies that receives the heat necessary to keep the whole apparatus in operation. Of course the feed pipes, F-1, 2 and 3 and the discharge pipes are all supplied with suitable valves for the regulation of the flow of the sweet-water. Means are provided for the steady removal of condensations; from calandria 1, by means of a steam trap and from calandria 2 and 3 by suitable pumps.

When the triple is to be set in operation, the water is turned on to the condenser and the vacuum pump started so that a partial vacuum is obtained in E-1, 2 and 3; valves F-2 and 3 being open. Valve F-2 is to be shut as soon as a vacuum of say 10 to 12 inches has been reached and F-1 opened taking in sweet-water. Presently F-2 is again opened and the inflowing sweet-water allowed to find its determined level (L) in the three effects.

If steam be now turned on, it will rapidly heat the sweet-water in E-1 and shortly cause it to boil, the vapor passing upward and through Vp-1 into C-2 where it is at once condensed giving up its energy in the form of heat to the sweet-water in E-2. Similarly E-2 reacts upon E-3 and the condensations in C-2 and 3 are withdrawn as they are formed.

PRINCIPLES OF MULTIPLE EVAPORATION

Leaving, for awhile, the condition of the effect as just established, a brief digression on the principles underlying multiple effect evaporation will clarify the phenomena within the effect in actual operation.

The principles of ebullition and evaporation, in so far as it is necessary to consider them here, may be briefly stated thus: The boiling point of a liquor of definite chemical composition, such as water, alcohol or ether, is constant under constant atmospheric pressure upon its surface. If the pressure be lowered, the boiling point falls; if it be increased it rises. The vapor rising from a boiling liquid is of the same temperature as the boiling liquid. Water, at normal atmospheric pressure boils at 212° F., and the vapor or steam rising from it is likewise at 212° F.

With these points in mind, consider the accompanying sketch as representing two flasks (a and b) of equal capacity and as containing 100 ounces of water each and at 32° F. The neck of the flask "a" is turned over and dips into the water in "b." The gas flame (f), heats the water in "a" to 212° F., and ebullition begins. The boiling is continued until the steam vapors pass through the tube-neck of "a" into the water in "b." At first the bubbles of steam issuing from the orifice of the tube will be rapidly absorbed, but eventually they will raise the temperature of the water in "b" from 32° F. (its initial temperature be it remembered) to 212° F. The source of heat is now removed and the residual water in "a" is measured and the augmented amount in "b" is also determined.

As a matter of fact, it will be found that the water in "a" has diminished by something like 20 ounces, (Actually 18.75 ounces) and the water in "b" has increased by that amount. Roughly speaking, and quite serving the purpose of this argument, one-fifth of the water in the *form of steam* from "a" has been able to heat five times its weight of water in "b" from 32° F. to 212° F. But if 20 ounces of water at 212° F., instead of steam, had been added to the 100 ounces of water at 32° F. in "b", the temperature of the mixture would have been only *62° F.* Evidently then, water in the form of steam at 212° F. contains much more energy than boiling water in liquid form at 212° F. This energy, not indicated by the thermometer but resident in the steam has been somewhat inaptly called "latent heat," but is really heat that has been transformed into another form of energy (kinetic energy) and reappears as heat energy when the steam from "a" is condensed in "b," as illustrated in the experiment.

If now we refer back to the effect and regard the calandria C-1 as the source of heat (f in the experiment), and let "a" equal E-1, and "b" E-2, and regard Vp-1 as the connecting tube between "a" and "b," we have a replica of the little apparatus. What happens then if steam be turned into the calandria C-1? The sweet-water presently begins to boil, and after one-fifth of its weight in the form of steam has passed over into C-2 and has been condensed there, the sweet-water in E-1 and E-2 will be at equal temperatures, *i. e.*, at their boiling points. We are now ready to return to the triple effect for actual operations.

A TRIPLE EFFECT IN OPERATION

Going back to the effect we find that it has been left charged with sweet-water and with steam turned into C-1. But upon the principles explained, the vapors of the boiling sweet-water in E-1 pass into C-2 and in their turn start ebullition in E-2 because, be it remembered, a slightly higher vacuum, hence a lower pressure, had been created in this body before the sweet-water had been drawn into it, and therefore, the sweet-water in E-2 will boil at a lower temperature than that in E-1, since the boiling point of a liquid falls in proportion to the diminution of the pressure upon its surface. Again, as the vapors pass from E-2 into C-3 the same phenomenon appears, in fact it is somewhat accentuated as the vacuum pump and condenser very quickly raise a comparatively high vacuum in E-3, something in the neighborhood of 27 inches of mercury. Therefore the sweet-water in E-3 boils rapidly and at a low temperature, say 120° F.

In a very short while the entire apparatus adjusts itself to a regular and continued evaporation so that as the boiling goes on the vacuua in the three effects will be about 5, 15 and 28 inches respectively; the approximate boiling points 212°, 180° and 120° F. will be observed in the effects E-1, 2 and 3. In fact, under a steady steam pressure in C-1, these conditions will vary but slightly at any time and it requires but the attention of the attendant to keep the density in the respective bodies within a certain prescribed or determined limit to secure a satisfactory concentration.

STEAM ECONOMY AND TRIPLE-EFFECT EVAPORATION

But why multliple-effect evaporation and not single-effect evaporation? Why complicate the apparatus? Because in discussing the phenomena of the boiling water in "a" and the effect of the vapors passing therefrom into "b" and there condensed, it was shown that these vapors contained not only the energy of heat as indicated by the thermometer, but also a great amount of kinetic energy not indicated by that instrument. Obviously if all the evaporation were done in a single effect not only all the heat but also all the kinetic energy would be lost in the condenser. But if the last body of a triple effect does approximately one-third of the work, then only one-third of the total energy will be thus lost, and the economy of the triple or quadruple evaporation is established.

While the general operation of a triple effect requires little supervision, there is one important point in particular that should receive systematic attention. The sweet-water from the char-filters invariably has an acid reaction, while those from the presses are alkaline. When these sweet-waters are mixed before entering the effects, the mixture may be either acid or alkaline—possibly neutral. Now alkaline sweet-waters tend to froth, while acid reaction is particularly objectionable as considerable inversion of sucrose may be induced during the concentration, especially in the first effect where a high boiling point is always to be observed. Only neutral sweet-waters should, therefore, be allowed to enter the effects.

Lastly it may be necessary to know (a) the total weight of water that is to be evaporated in a given time and (b) how much water must be evaporated in each effect in order to maintain the balance of work in each element of the effect. It may be a question of running the effects under maximum capacity, for instance, or to determine how long a time it will take to concentrate a certain quantity of sweet-water from and to a certain density.

Under (a) the question will be: How much water must be evaporated when the light sweet-water is at "b" deg. Brix, and is to be concentrated to heavy sweet-water at "B" deg. Brix? This can be determined by the formula,

$$\mathrm{CW}\left(\frac{\mathrm{B}-\mathrm{b}}{\mathrm{B}}\right)=\mathrm{x}.$$

in which,

C = Volume of light sweet-water in cubic feet.
W = Weight of cubic foot of light sweet-water.
b = Degree Brix of light sweet-water.
B = Degree Brix of heavy sweet-water.
x = Pounds of water to be evaporated.

Illustration: Suppose there be on hand for evaporation 1,000 cubic feet of sweet-water at 12° Brix, to be concentrated to 54° Brix, then $C = 1{,}000$; $W = 65.37$ pounds per cubic foot; $b = 12$ and $B = 54$.

Therefore,

$$1{,}000 \times 6{,}537 \left(\frac{54 - 12}{54}\right) = 50{,}843.$$

If the effects are able to evaporate 12,000 pounds of water per hour, then:

$$\frac{50{,}843}{12{,}000} = 3 \text{ hours and 30 minutes, approximately.}$$

We know, then, that under the assumed conditions we have 50,843 pounds of water to evaporate and that this can be done in a little under 3½ hours.

But in any effect system each effect must approximately evaporate its proper quantity of water. When this does not occur, or cannot be maintained the system is not balanced. This want of balance may be due to several causes; to poorly functioning air or tail pumps; to scale on the calandria; leaking joints, etc. In any event it is advisable to be informed what the density should be in each effect. Knowing the density of the sweet-water entering the first effect the density that should be maintained can be determined.

Assuming the same basal figures as in illustration (a), the question under (b) can be answered by the following method:

Under (a) it was determined that the water to be evaporated was 50,843 pounds, therefore each effect should evaporate

$$\frac{50{,}843}{3} = 16{,}947 \text{ pounds.}$$

In 1,000 cubic feet of sweet-water at 12° Brix there are 7,840 pounds of solids and 57,530 pounds water.

Therefore if I effect evaporates 16,947 pounds water and sweet-water enters at 12° Brix

$$57{,}530 - 16{,}947 = 40{,}583$$

$$40{,}583 + 7{,}840 = 48{,}423$$

$$\frac{7{,}840 \times 100}{48{,}423} = 16.19^\circ \text{ Brix}$$

must be the density of sweet-water maintained in this effect.

II effect receives sweet-water at 16.19 Brix and 16,947 pounds water are there evaporated, then,

$$48{,}423 - 16{,}947 = 31{,}476$$

$$\frac{7{,}840 \times 100}{31{,}476} = 24.9^\circ \text{ Brix}$$

must be the density of sweet-water maintained in this effect.

III effect receives sweet-water at 24.9 Brix and 16,947 pounds water are there evaporated, then

$$31{,}476 - 16{,}947 = 14{,}527$$

$$\frac{7{,}840 \times 100}{14{,}527} = 53.96^\circ \text{ Brix}$$

the density required.

It follows then that when a triple effect is concentrating sweet-water from 12 Brix to 54 Brix that the densities in the effects should approximately be maintained at respectively 16°, 25° and 54°, as shown below for varying original densities.

Orig. Br.	Br. leaving I effect	Br. leaving II effect	Br. leaving III effect
10.0	13.7	21.9	54.0
12.0	16.2	24.9	54.0
14.0	18.6	27.7	54.0
16.0	21.4	30.1	54.0
20.0	25.4	35.0	56.0
15.0	19.8	29.1	55.0
10.0	13.8	22.3	58.0

REMELTS

Remelts from Raw and Refined Intermediate Products.—The sweet-water from all sources having been disposed of as described in the preceding paragraphs, they reenter the operations of the house, mingled with greens and raw liquors as their purity may suggest.

Consulting Diagram VIII, it will be seen that in the "Raw House" the operations move along three systems (R, A and B). In Chapter I, Part II, affination, pressing and bag-filtering of raw liquors and syrups has been dealt with, so that the methods in systems R and B only remain to be considered. In the diagram it will be seen that AS, RS, RW and BSS are indicated as going "to filters," *i. e.*, by way of presses or bag-filters to the char-filters. BSS and RW are usually united, as also AS and RS, thus constituting raw syrups and raw liquors. Again AG may be used for three purposes, according to circumstances, *i. e.*, it may be returned to G storage tank or used either for raw remelt or for black-strap boiling. AW may also on occasion be used for other purposes.

Raw Remelts.—When the maximum production of granulated is sought from the refining of a given raw sugar, it is necessary that as much high-purity liquors shall be obtained as possible, and this can best be brought about by a series of recrystallizations of greens and washings. The same practice is followed when a limited amount of softs are required, as shown in the diagram. AG and RG are used for this purpose by being sent to either the R or BS vacuum pans.

But remelt boiling has usually a twofold object: the production of a washed crystal yielding a high purity liquor on the one hand, and syrups and washing that will be serviceable for subsequent boilings on the other. The composition, therefore, of the massecuites are calculated to yield the required syrups and washings; the liquors coming from such massecuites are accepted as they may come. The greens from the affination can be easily maintained at a purity required for the remelt massecuite, and further, the charges for the pan can be adjusted by the addition of either AW or RW, should the greens be too high or too low. The charges, then, having been prepared and sent to the pan, the boiling proceeds as described in the chapter on Production, and the discharged massecuite is purged and washed and the dissolved sugar and washings sent out of the system, eventually reaching the char-filters.

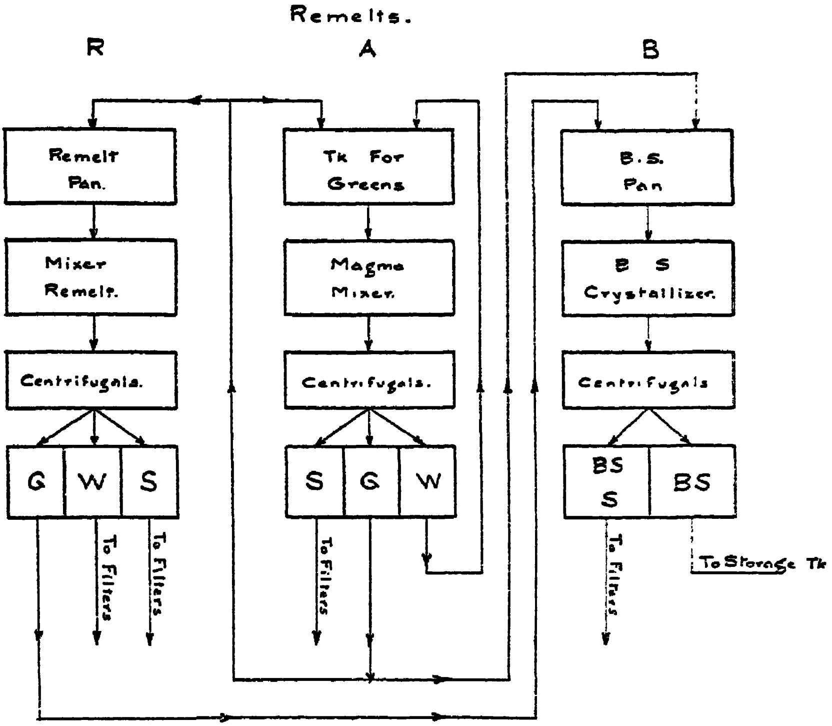


Diagram VIII.

Refined Remelts.—As in the raw house so also in the refinery, intermediate syrups are obtained, that cannot be directly used for any of the products required. These, then, must be reboiled to secure liquors and syrups of desired purity for further manipulations.

Such intermediate material will be granulated syrup of low purity and high color and syrups accumulating from soft-sugar boilings. In the case of granulated syrup, the purity that may be required for remelts is readily controlled, since the syrups from any granulated strike may be directed to any convenient storage tank to await remelt boiling. The process here is quite similar to that followed in the raw house. The washed sugar obtained from such a refined remelt boiling is, of course, of high purity (98 to 99.5) and sufficiently devoid of color to be melted and turned into the liquors from the char-filters destined for granulated sugar. The washings and syrup, however, may be sent to the char-filters from whence they return as "A" liquor (vide chapter on Purification) or once more boiled to obtain a final syrup suitable for End-Products.

END-PRODUCTS

From Diagram VIII it may be seen that the greens (AG and BG) from the affination and remelt work are sent to the BS pan to be reboiled for a very definite purpose, *i. e.*, the production of final molasses. Now the chief aim of all the operations incidental to section B is to secure as low a purity EP as possible at this station. As indicated, the BS going out of the house as EP, may have a purity of from 35 to 40, and under certain conditions may even fall to an apparent purity of 30 or a little under that figure. If then, it be assumed that the final BS has a purity of 40, the sucrose and non-sucrose will be present in the proportion of 2 and 3 respectively in the dry substance of the BS. But as the ratio of sucrose to O. N. S. in the AG and RG are respectively 4 to 1 and 3 to 2, it is evident that considerable sugar should be extractable from these products, and it is to this end that such intermediate greens, as AG and RG are gathered and sent to the B system to be boiled, crystallized and centrifuged with resulting BSS and BS.

Of course it is frequently important to estimate how much BSS and BS will be obtainable from a given quantity of greens at a known purity. This can be determined by the formula $\left(\frac{P - p}{S - p}\right) 100 =$ per cent BSS on solids, in which $P =$ purity of G; $S =$ purity of BBS and $p =$ purity of BS.

Illustration. Let it be assumed that the mixed greens destined for the BS pan have a purity of 70 and that the BSS purity will be 90 and the BS purity 40, then

$$\left(\frac{70 - 40}{90 - 40}\right) 100 = 60.$$

Every 100 dry pounds of this green syrup will then yield 60 pounds dry BSS and 40 pounds dry BS, or 50 pounds black strap containing 20 per cent water.

Problem of Obtaining Low-Purity End-Products.—It is a well established fact that crystallization of sucrose from syrups of low purity is more difficult to induce and to maintain than in syrups of higher purity, indeed, generally speaking, the facility of pan work in this respect decreases as the purity falls in the solution from which crystals are to be grown. Logically we expect, and indeed find, that at a certain purity no further crystallization of sucrose will occur under any now producible conditions, and also, that much above the minimum purity, special methods must be used and efforts made to induce crystallization in ordinary pan work. And it is for these reasons that certain salient features of the physical and chemical properties of final molasses must be discussed.

As will presently appear the purgings from a BS massecuite may not infrequently have the following composition:

	Per cent
Sucrose	32.0
Reducing sugars	23.0
Ash	10.0
Organic non-sucrose	20.0
Water	15.0

indicating, as pointed out on page 48, that we have in such a case a viscous, tenaceous, semi-fluid solution contradicting the laws of pure sugar solubility. But in truth, such last products can hardly be regarded as solutions at all, rather may they be presented as complex mixtures of intercombined compounds of cane-sugar, invert sugar and a large number of organic acids with the bases of potash, soda, and lime. And moreover, the composition of the 20 per cent of O. N.S. must vary in many respects in accordance with the different juices and processes of extraction practiced on the sugar estates. And, lastly, the molasses film on the raw crystals is the adhering molasses from which the crystals have been spun, therefore the RG from the remelts must be a somewhat similar product, and it is from this kind of greens that the final low product of both mill and refinery are eventually obtained.

PRESENT AND FORMER METHODS IN FINAL MOLASSES PRODUCTION

Before the introduction of crystallization in motion, presently to be described, raw molasses and also low-purity greens of the refinery were boiled "blank." Blank boiling and subsequent tank crystallization consisted of reducing any suitable molasses, having a purity of from 55 to 60 to a density low enough to dissolve any small adventitious crystals and drawing this diluted material into a vacuum pan. At this purity crystallization will not take place in the pan, even though the concentration be carried to 93° or 95° Brix; the concentrate acquires a consistency of a very heavy syrup and is without grain when discharged from the pan at the density mentioned. From the pan the "blank" was dropped into crystallizing tanks. These tanks were vessels of a capacity to hold the contents of a pan and were usually kept in a moderately warm room, about 100° F. In 2 or 3 days crystallization began, but 12 to 14 days were necessary to complete the process, *i. e.*, to reach a molasses purity between 35° and 40°. After the blank had "ripened" the contents of the tank were sent into a mixer and the molasses purged from the crystals in the usual manner.

The evident cumbersomeness of this blank method lies in the fact that a large number of tanks had to be provided and considerable floor space allowed for their accommodation. Even under a moderate production of refined or raw molasses nearly 1,000,000 pounds of such blanks would be in various stages of ripening and consequently so much material held from immediate production.

But the unpleasant obtrusion of the "molasses question" upon the routine of production and refining, and the endeavor to recover as completely as possible all practically crystallizable sugar from the intermediate products, gave a marked impetus to research in the problem of syrup desugarization. Investigation on the chemistry of what may be called "molasses formation," went hand in hand with practical experiment in cane and beet sugar factories, resulting finally in the general practice of "crystallization in motion."

The natural inference was, that a thorough study of the chemistry of molasses formation might eventually lead to a considerable reduction of the sugar content in the final molasses. But despite many years of investigation by prominent, scientific and practical men the final word upon this question has not yet been uttered. Persistent endeavors however, have lead us to the very efficient process just mentioned. It was always the question: What produces molasses? Invert sugar, mineral salts, combinations of invert sugar with metalic bases, viscosity, etc., have all in their turn been assigned as the cause of molasses formation. But we now know definitely that viscosity is one of the important factors in the case. If, therefore, we can reduce the viscosity, or counteract its effect, we can, and do obtain a crop of crystals and a batch of low-purity molasses in from 36 to 60 hours, which formerly required from 12 to 14 days under the system of blank boiling.

During the early days of the practical application of the principles of crystallization in motion, very special vacuum pans were devised; prolonged and involved methods of boiling suggested, and finally the crystallizers into which the massecuites were discharged, were provided with vacuum appliances. All these precautions were considered as contributing to the greatest efficiency of the process, but the simplified system now generally used promises to be quite as serviceable. Furthermore, the original tendencies were to drive the sugar extraction to extremes, seeking to secure a final molasses of 25 to 27 apparent purity. Indeed, such figures were obtained, but whether the few extra pounds of sugar secured were economically obtained is very doubtful. Unquestionably in many cases the low purities were due to inversion and *not* to recovery of sucrose.

Crystallization in Motion.—A grasp of all the steps of this process and the principles involved may best be obtained by following an illustrative operation through its different phases, the main object being a final molasses of 40 purity or lower.

I. Preparation of Material to be Boiled.—As distinctive of blank boiling, the material destined for crystallization in motion is kept at a higher purity because a fairly strong grain is to be grown in the pan. Usually two charges are prepared somewhat along the lines described under soft-sugar boiling.

The first charge may be any mixture of raw liquor and syrup, bringing this charge to a purity of, say 82. The second charge may be made up from any remelt greens or any lower-purity material available. Of charge one a quantity is drawn into the pan to form a volume of liquor in which sufficient crystals may be started and eventually grown by the periodic introduction of charge two as required in the usual boiling practice. This second charge, furthermore, should be reduced to a density of 50 Brix, or even lower if the purity be below 60. The entire composition of the material should be so proportioned that the massecuite at striking shall have a purity of 65 to 70. It is to be noted that while the boiling is going on, there is a gradual decline of purity of the molasses surrounding the growing crystals; firstly, because as the crystals grow, sugar is being extracted from the mother-liquor and deposited on to the crystals, and secondly, the introduction of the second charge syrup, further contributes to this reduction. It follows, then, that as the boiling proceeds, and especially towards the end, crystallization is going on in a liquid of low purity and therefore of high viscosity.

II. Condition of the Massecuite in the Vacuum Pan.—Attention must at once be drawn to the physical properties of low-purity sugar solutions. On page 48 it is shown that a molasses of 53 purity (polarization 45) and 15 per cent water content will hold 50 per cent more sugar in solution than the law of pure sugar solution provides for. Besides this supernormal sugar in solution there are also present 2.6 parts of non-sugar substances. In brief, such a molasses or low green may be regarded as a supersaturated solution and indeed, all low-purity syrups of high density are supersaturated solutions from this point of view. Inferentially, then, it may be assumed that the liquor of 82 purity, in this illustrative case, will have to be boiled down to some point of supersaturation before any crystals will form. This deduction is borne out in practice, and crystals do not form until a certain supersaturation has been reached. But crystallization having once been induced and continued through the hours of boiling by the growth of the crystals, pure sugar is extracted from the mother-syrup, progressively lowering its purity so that as the operation proceeds a progressively higher supersaturation must be maintained. But greater concentration means greater viscosity, which in its turn means greater immobility of the syrup around the growing crystals. Increased concentration, viscosity and immobolity then, together with the falling purity, consequent upon the extraction of sucrose by the growing crystals, will presently bring about a condition under which any practical attempt at further crystal growth would be futile. At this stage of the boiling the massecuite will be composed of a mass of pure crystals surrounded by a dense, viscous, immobile syrup, so low in purity that it cannot readily yield any more of its sucrose to the crystals, in other words, crystallization has become very slow, or may almost cease. But an analysis of the crystals and the syrup—now almost a true molasses—will show that if the massecuite were now centrifuged a crop of crystals—about 90 to 93 purity—would be obtained, while the molasses would still have a purity of 50 or more. Evidently the goal has not yet been reached. Again it will be found the the water content of the massecuite may lie between 6 and 7 per cent, and the molasses itself will contain not more than 12 to 13 per cent of water; much further concentration, therefore, would be out of the question.

III. Crystallization in Motion.—However, a further extraction of sugar *is* possible provided due provision be made to meet the best conditions under which crystallization or grain growth may continue. The old blank method we know would eventually yield the 40 purity molasses after standing in tanks 10 or 12 days. But if instead of dropping the strike into a tank and leaving it there, it be discharged into a "crystallizer," provided with a stirring device and a means of controlling the temperature, then the massecuite may be kept in a condition tending to expedite further crystal growth, so that after 36 to 48 hours the molasses will have fallen to the desired purity—the goal will have been reached in one-quarter of the time—and why?

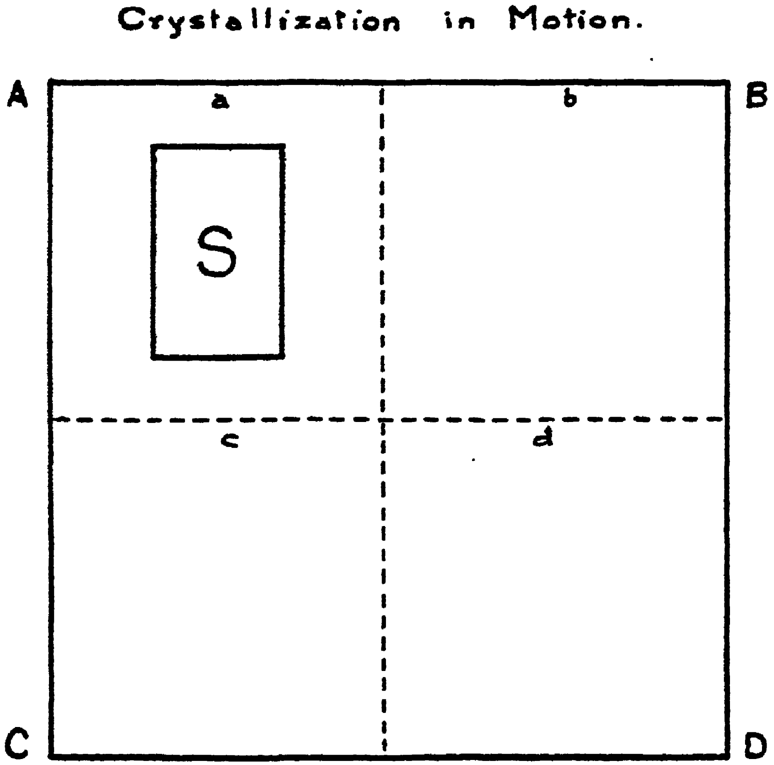


Fig. II.

This question can be answered by the aid of sketch, Fig. II, in which ABCD represents a cube of molasses and S a crystal of sugar. S has a purity of nearly 100, ABCD, the molasses, is at 50 purity. Not forgetting the chemical and physical properties of this molasses, we must again postulate that it is exceedingly viscous, immobile and near the point of its minimum practical purity. What would be the prevailing condition if in this system the crystal S were to remain undisturbed in section "a" of the four sections a, b, c and d? By reason of viscosity, immobility is pronounced and the sphere from which S might draw further molecules of sucrose to its surface would be limited—let it be assumed—to the sucrose available in the molasses in section "a." As this molasses in "a" gives up its sugar to the crystal S, the molasses will fall in purity, reach 40 and crystallization, or the growth of S will cease. But why will not the available sucrose remaining in sections b, c and d aid in the continued growth of S, until b, c and d are in their turn exhausted? Given sufficient time the molasses in b, c and d would yield their sucrose to S. But for purpose of argument S is at rest, and as it remains in section a, it exhausts that section of its sucrose, and in so doing it lowers the purity making the molasses still more viscous, still more immobile, building around itself, as it were, an impenetrable sphere of molasses which is unable any longer to supply sucrose to the crystal S. At this stage the average purity of ABCD would have fallen to 47 only, since "a" will have reached 40 and b, c and d still at 50 purity.

It may be imagined, however, that the molecules of available sucrose remaining in sections b, c and d might fight their way through to S; and this may be conceded they will do by reason of their molecular activity and the molar motion within ABCD, but their progress would be, and is comparatively slow as shown practically in blank tank crystallization. Indeed, we know that such a molecular struggle goes on for nearly 2 weeks, before the last sucrose molecule has lodged itself with its kin upon the crystal S. Again, it may be imagined that if some intelligence were to take S out of section "a" at the moment of exhaustion and carry S at proper successive intervals from b to c and then to d, that the delayed crystallization would be avoided. This in effect is actually done by mechanical means by the stirring device of the crystallizer. By a constant, but slow stirring, S is continuously carried from a to b, c and d and around in this cycle until the entire molasses is exhausted to within practical limits. This is to say, that instead of having a progressively retarded crystallization going on within "a" only, and then very slowly from b, c and d to S, as in the tank method, we have it in the motion method going on in a, b, c and d in rapid succession, *i. e.*, in four times the volume of the circumambient molasses, and therefore crystallization is four times as rapid.

In the application of these principles, the mechanical stirrers are rotated at the rate of three revolutions per minute, or even less, and the temperature of the massecuite should be regulated to meet the observed rate of crystallization; but this is seldom, if ever, scientifically followed in practice as the labor of supervision would hardly pay for the extra yield secured. However, some attention is required and the adjustments, if necessary, may be made empirically. Generally speaking, there should be a declining temperature, maintained or induced, for the reason that as crystallization goes on, the sugar extracted from the molasses reduces the density somewhat and, therefore, by lowering the temperature the critical point of crystallization is compensated for.

A strike of this kind is usually discharged from the pan at 155° F., and the massecuite allowed to gradually fall to 135° F., or thereabouts.

IV. Centrifuging of Black-Strap Massecuite.—Under a special system of crystallization of low products, the massecuite is discharged into a crystallizer, Section 1, as shown in the sketch, and the succeeding strikes into the same compartment. Strike 2 pushes the major part of strike 1 into the compartment 2, and so on until the five compartments have been filled and the purging of the black strap may begin; compartment 5 only being pro-

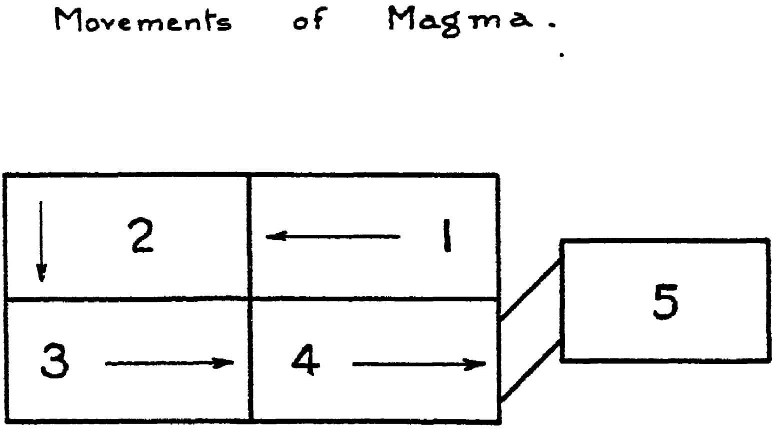


Fig. III.

vided with centrifugals. Compartment 5 is water-jacketed so that in case of a specially viscous massecuite, hot water may run through the jacket and the mass raised to any permissible temperature to facilitate centrifugal work. One hundred and fifty degrees may be allowed as the viscous molasses will not readily dissolve the grain, especially if it is of fair dimensions.

At best, however, centrifuging of black-strap massecuite is slow and the resulting sugar, dark and seldom above 90 purity by reason of adhering molasses.

The black-strap sugar is melted, mingled with RW, pressed or bag-filtered and sent to the char-filters.

The black strap as it comes from the machines may have a density of 85 to 88 Brix and for that reason must be reduced to 80 Brix to facilitate subsequent barreling.

Medium and low-grade table syrups may, of course, also be considered as end-products, but the discussion of these will be treated under Special Products.

RAW AND REFINED END-PRODUCTS

Table or Barrel Syrups.—The black strap spun from the massecuite may be regarded as a raw end-product in distinction to a refined end-product, obtained by the boiling and crystallizing of mixtures of granulated and soft-sugar syrups by methods quite similar to those described under black strap boiling. The sugar obtained from such a massecuite is usually sent to the char-filters—preferably over a medium grade char—and returned to the refinery as liquor for soft sugar. The syrup spun from such a massecuite is set aside as a basic material for the subsequent production of any grade of table or barrel syrup required. This crude table syrup is never as dark as the black strap, but not infrequently lower in purity, due to the presence of a larger proportion of invert sugar.

From a stock, then, of black strap and crude table syrup differently refined table syrups may be made by mixing the available stock in proportions necessary to produce the article required. No definite rule can be laid down for such mixtures, as the selection to be made will differ for each lot, since the raw material is seldom uniform. If, however, a specific quality has been determined upon, and a large and continued production is possible, a uniform and standard article can be turned out b providing a steady and maintained line of operation.

Although black strap offers difficulties in syrup production and, at best only a very dark syrup (resembling treacle) can b produced, it may be well to provide means for refining this material by itself without any admixture of crude table syrup.

The initial difficulty will be encountered in the endeavor to clarify the water diluted black strap. This is so heavily charged with non-sugar substances and exceedingly fine suspended matter, that attempts with the ordinary methods of clarification always fail. Neither diatomaceous earth nor phosphate paste will efficiently aid either press or bag-filter in the work of holding back the copiously suspended impurities. Filter-press work will eventually yield a syrup clear enough to send to the char-filters, but the capacity of a press is so greatly reduced that few refineries are equipped with a sufficient number to meet the demand of only a limited output. Nor, indeed, would such a method pay, since the washing, cleaning and general work involved in the handling of so many presses would draw heavily upon the labor cost. However, under Special Products a process will be described which has avoided the difficulties here indicated.

Crude table-syrup material, on the other hand, can be more easily dealt with. If table syrup is to be produced, the crude syrup may be reduced to 50 or 53 Brix, sent to tanks for treatment with "earth" or phosphate paste. The quantity of the reagents to be added will depend upon the particular batch of syrup under treatment, and can best be determined by a laboratory trial. As a rule fairly clear liquor will be obtained, although very little of it will be brilliant. The slight cloudiness of the average will be corrected, however, if proper precautions be taken during char-filtration.

Char-filtration and Concentration of Table Syrup Liquors.—As may be expected, char-filtration of the syrup liquor produces very marked changes, physical and chemical, on such solutions passing through this medium of purification, but at the same time the char rapidly loses its power, so that if high color reduction be aimed at, two or even three times the weight of char to the syrup solids passing through it must be employed. An important matter in any event is the control of the syrup before it is allowed to enter the filter. Certain conditions must be present, or be established, if the full effect of a given quantity and quality of char is to produce its maximum result.

Density, temperature and chemical reaction are the most important.

I. Density.—Liquors and syrups of high purity are to advantage filtered at 55 Brix, at 180° F., but table-syrup liquors should not be put upon the char under 58 Brix, at 180° F., and if the char is free from fine grain, the density may, with advantage, be raised to 60 Brix at 170° F.

II. Temperature.—As all grades of table syrups contain a large amount of invert sugar—from 15 to 30 per cent—as low a temperature as possible should be maintained throughout all operations. Invert sugar is prone to form decomposition products with some of the metallic bases, particularly with calcium, sodium and potassium, each of which gives rise to a reddish brown color, which the char cannot remove, or to a slight degree only.

III. Reaction of Syrup Liquors.—Generally speaking, it is more difficult to obtain brilliant char filtrates when liquors and syrups of low or medium purity, run with acid reaction from the char-filters. Neutral or slightly alkaline filtrates are usually sparkling although they will be somewhat darker than similar filtrates of acid reaction. On the other hand, undue alkalinity must be avoided both in respect to solutions entering or leaving the char-filters. Particular precaution should be exercised in this direction when table-syrup liquors are to be filtered, and it will always be desirable that rather a low alkalinity characterize the liquor entering the filter than that it should issue with alkaline reaction. This condition indicating that during the entire period of filtration the solution in question must have been alkaline to a considerable degree, and therefore subject to the formation of those brown compounds mentioned in the preceeding paragraph.

While for ordinary purposes, acid reaction may be corrected by the addition of lime, table-syrup liquors should be brought to the desired neutral or slightly alkaline reaction by the addition of carbonate of soda. The soda may with advantage be added to the liquor while it is being prepared for press-filtration, and in such quantity that a faint, though not distinct alkaline reaction, as indicated by litmus paper, shall be apparent as the liquor flows from the press. Sodium carbonate has the further advantage over lime, that it may be used to correct acidity at any stage of the process.

If density, temperature and alkalinity be carefully regulated, and the liquor conform to these three conditions a brilliant table syrup will result.

IV. Concentration.—The table-syrup filtrates may be run for pan concentration as long as the filtrate remains clear, but as soon as the sweet-water gives sign of "smokiness" it should be turned back for diluting the next oncoming batch of raw syrup. By this method a constant supply of clear filtrate may be drawn into the pan for concentration.

Concentration is, of course, nothing more than a boiling down of the char-filtrates to the required density of the finished syrup. Two conditions are, however, to be sought, *viz.*, a high vacuum and a low-boiling temperature; these are interdependent and the one ensures the other. But comparatively low vacuum and consequent high-boiling temperature, in most cases implies a leaky pan, which means that air bubbles may be continually passing through the boiling syrup. Now low-purity table syrups are very prone to oxidation of their non-sugar organic compounds with the production of a disagreeable brown coloration, so that the importance of concentrating under the best possible conditions become very evident and should be given close attention.

The prevailing boiling point of syrup during concentration should be between 120° to 125° F, and if the finished syrup is let out at that temperature, the density should be between 77.5° and 79° Brix. The higher density should be reached in summer syrups only, unless required for some special purpose.

V. Use of Sodium Carbonate and Sulphur Dioxide.—Occasionally light brown or dark yellow syrups are in demand, and to meet this, the syrup filtrates must be "sulphured" with sulphur dioxide fumes. This can be done in the usual way as described under soft-sugar production. It is advisable never to exceed 300 milligrams of the gas per liter of filtrate.

Sodium carbonate may sometimes be used to correct a slight "cloudiness" of a syrup filtrate. If this cloud or smoke appears in the filtrate at the middle stage of the filtration, or when the pressing-off water begins to perceptibly reduce the density of the filtrate, such a condition does not necessarily unfit the filtrate for direct concentration to a finished table syrup. Indeed, the clouding of the dilute portions of such filtrates is not at all uncommon and is due to the fact that sugar solutions above 55 Brix are solvents of many complex organic salts of sodium, potassium and calcium, salts that are less soluble in dilute than in concentrated sugar solutions, hence precipitation occurs when char-filters become diluted by the pressing-off water. Or, perhaps, another view may be taken as to the clouding. It may be that the dilution of the liquor brings about a decomposition of these metallic salts and that the so-decomposed products are insoluble and are therefore precipitated in the form of exceedingly fine particles to which the cloudiness is due. However this may be, there are two distinct cloudings to be observed in the filtrates. One is due to the precipitation of the organic salts just referred to, the other, a distinct decomposition product, is "reverted" phosphate of calcium. The cloud due to the phosphate can readily be distinguished from the organic precipitate by the fact that on further dilution of the liquor and heating it to near the boiling point, the precipitate will presently become flocculent. Also upon slight acidification of the liquor with hydrochloric acid the precipitate disappears immediately, while the precipitate due to the organic substances is insoluble in the acid and will not yield a clear solution even after prolonged heating.

If, however, sodium carbonate be added to very slight alkalinity, the organic precipitate dissolves and a clear, even brilliant solution will be obtained. A distinct darkening in this case is always to be observed, but in the lower grade of table syrups this does not at all interfere with its attractive quality, and, indeed, it improves the flavor, thus achieving in this case the practice of adding common table salt to counteract the astringent taste due to lime salts present.

Finally, then, a slight cloudiness of syrup-liquor filtrates does not necessarily mean a cloudy finished product. Not infrequently this cloud disappears as the liquor is being concentrated in the pan, and if it does not, it will in most cases, yield to the action of sodium carbonate. This is added to best advantage when the syrup has reached a density of about 60° Brix. The soda should be added in small doses and the effect watched after each addition. No particular formula can be given in this respect, but with a knowledge of the reaction of the liquors, be they neutral or acid, satisfactory products will be assured.

PART III

CHAPTER I

ACCESSORY MATERIALS, ADJUNCTIVE OPERATIONS AND CONTROL

INTRODUCTORY

The series of operations described in Part II frequently required some reagent or accessory material as a factor in the attainment of the immediate or remote object of a particular phase in the course of refining. Sometimes such a reagent or such an accessory material is manufactured or prepared upon the premises, or if not, it may be necessary to exercise special care in its application, and if it is recoverable for subsequent use, definite steps in the process will have to be followed involving adjunctive operations incidental to the main refinery processes. From time to time different materials have been suggested, experimented with and used practically in this or that department of mill, factory and refinery. Some idea may be formed of the area of circumspecting research when it may be stated that some six hundred substances have been proposed and applied to some of the steps in sugar production. From cane or beet field to the final phase of refined products the engineer and chemist have sought either to improve, aid or economize production by some accessory material of promise. Needless to say that not more than 10 per cent of the six hundred proposed substances have proved of practical value. Some have maintained themselves for awhile and have been superseded by others more economical or better. Many of the more fanciful kinds were tried and discarded after shorter or longer periods of practice, and, finally, many have not gone beyond laboratory experimentation.

Of course, such materials as coal, fuel-oil, lubricants, water, steam, metals and compounds of all kinds used by the engineer and the mechanic will not fall into this category and are related to production in a remote sense only. Engineer and chemist will jointly determine the qualities best suited to the particular requirements of the plant in such cases.

Control is connected with accessory material and adjunctive operations in so far that the handling of certain reagents and materials not infrequently play an intimate part in the interrelation of the departments and may effect the general economy of the institution.

ACCESSORY MATERIALS

With the exception of bone-black or char, refiners as a rule do not occupy themselves with the production of accessory materials, indeed, they are so few in number, and most of them require special appliances and processes for their production that in some instances virtually subsiduary establishments would have to be maintained to meet the demands of even a moderately large sugar house. Beet sugar factories nearly always, and cane estates frequently burn their own lime-stone, but beyond this the production of accessory material seldom goes.

Nor is the list of such accessory materials a long one and may be set down as comprising:

1. Quick Lime.
2. Diatomaceous Earth.
3. Hydrochloric and Sulphuric Acids.
4. Phosphate Paste and Phosphoric Acid.
5. Sulphur.
6. Sodium Salts.
7. Fabrics.
8. Carbonic Acid.
9. Decolorizing Blacks.
10. Bone-black.
11. Raw Bones.

1. Quick Lime and "Milk of Lime."—This material is very extensively used in the manufacture and refining of sugar. In the refinery its chief use is that of a neutralizing agent. As has been indicated, nearly all raw cane sugars have an acid reaction, and as even organic acids have a decided invertive power, which increases with the temperature at which any sugar may be dissolved, it is necessary to neutralize these acids by the addition of slacked lime, usually in the form of a "Milk of Lime."

Milk of lime is made by slacking quick lime in a manner similar to that used in making mortar. There are several lines of procedure to be observed, however, and the composition and general quality of the quick lime should be noted. Serviceable milk of lime can be made only from a quick lime that has been properly burned and is reasonably free from objectionable impurities.

Laboratory examination and analysis will, of course, determine the choice of one quick lime over another, but inspection by superintendents of lots or shipments as they arrive should never be neglected, as any irregularity at a remote lime kiln, or any variation of the lime stone will effect the quality of the lime delivered.

Gritty and underburned lime can frequently be discovered before the lime has been slaked, but a better method of observing lime qualities will be to watch the action of the lime during slaking and by a daily examination of the quantity of sand and other residue from the lime tubs or any apparatus in use.

In refineries, where the quantity of lime used is inconsiderable compared with that employed in cane mills and beet houses, a simple arrangement for slaking the lime and for handling the "milk" may suffice. As the lime is almost exclusively used in quantity at or near the melters and treating tanks, it may be conveniently prepared near such equipments in the following manner:

The quick lime is placed in one section of the slaking tub, SS, and cold water run upon it in such quantity that after the chemical reaction has been completed the slaked mass shall be fairly plastic and with the addition of a little more water becomes thin enough to flow under agitation through the screen behind the gate, "g," into the milk box, M. While the first batch is "seasoning" in the first section, a second batch is similarly prepared in the second section; the operations in this manner alternating. It is always advisable to allow 3 to 4 hours' seasoning, as otherwise some lime particles may find their way into the finished milk.

The diluted paste from SS is run into the mixing tub, M, where the paste is reduced by a further addition of water to standard

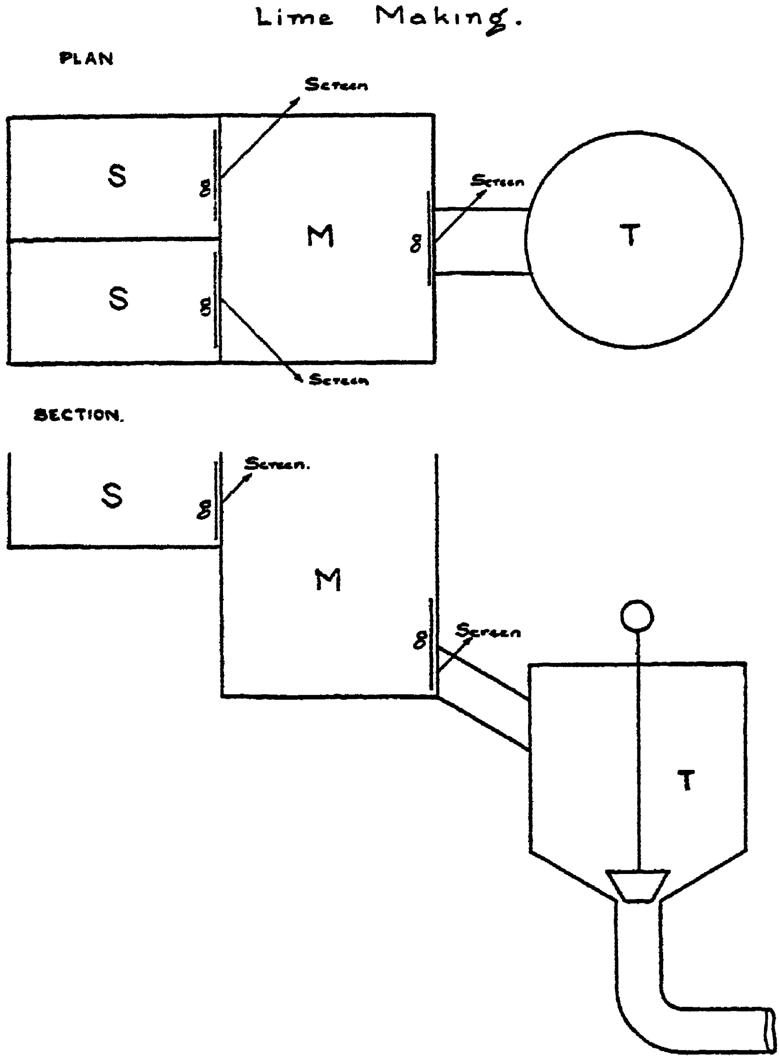


Fig. IV.

milk of lime density, usually about 20° Brix, or as custom may fix the standard. From M, the lime, now milk of lime is once more run through a finer screen—intended to hold back any insoluble particles that may have passed over from SS—into the distributing tub or tank, T. As this tank is essentially a storage tank it must be provided with a stirring device and a flushing plug, "p," to be drawn for periodic cleaning. When the milk from T is to be distributed to the different stations by a piping system, a circulating pump must be kept in constant operation to prevent settling of the undissolved slaked lime, for milk of lime is slaked lime in suspension, as only a small fraction of the calcium hydrate (slaked lime) is soluble in water.

Reverting once more to lime inspection, the quality of the lime may be roughly determined by examining the residues that accumulate in the receptacles. Undue quantity of residue will, of course, condemn the lime, while a small dash of diluted hydrochloric acid upon the sampled residue will indicate, if effervescence take place, that the lime has been underburned, that is, it contains undecomposed lime stone—carbonate of calcium.

Any insoluble, non-effervescent residue will indicate sand or clay. Sand, in particular, is objectionable, as finely divided particles of this will pass into the milk, enter the apparatus and machinery with consequent damage in many respects.

As regards the quality of lime acceptable for refining purposes, and what limits shall be set as to impurities, etc., the chemist will in most cases decide.

2. **Diatomaceous Earth.**—The function of this earth in defecation processes has already been described in Part II, page 59. The different proprietory names under which the prepared natural earths are offered for sale give no clue as to the value of the article as a filter-aid. Nor, indeed, is a chemical analysis of much significance; a simple physical examination will, in nearly all cases, be sufficient and indicate, at least, the relative efficiency to be expected from the contemplated use of one brand or another.

Color, Texture and Weight.—The most striking differences between earths will be their color, texture and weight.

Color.—This may shade from a cream to a brownish red. But the color of an earth is of very little importance since its depth is simply due to a very small percentage of iron oxide, which in no way interferes with the purpose for which the earth is to be used in refining. An earth of a light shade may not be as serviceable as a darker one or vice versa.

Texture.—By texture is meant the state of division of the diatoms and the absence of sand and clay, both of which are objectionable constituents. When comparatively free from sand, the earth will have a floury feel between the fingers and adhere tenaceously to them.

A rapid estimate of sand and heavy earthy particles can be obtained by shaking up a definite weight of the earth in a fixed volume of water. The rate of sedimentation and the quantity of sand, which soon separates from the diatomaceous particles, will give an indication of the purity of the sample, particularly if a comparative test be made against a standard product of known efficiency. The presence, to a marked degree, of sand and clay above that of a standard quality should be considered as an indication of an inferior product, and the filtering properties of such an earth would be comparatively poor and the brand should be rejected for that reason if for no other.

Clay can only be conveniently detected by the greasy feel it imparts to the material.

Weight.—If the specific gravity of an earth in bulk be taken, that is to say, if a cubic foot of the material be weighed, considerable variation will be observed. The range of earths will fluctuate between 8 to 33 pounds to the cubic foot. The comparative gravity in bulk may be obtained by shaking the earth into a cubic foot measure under a definite number of uniform taps. The so-determined bulk gravity will range from 0.120 of the lighter sort to 0.528 of the heavier kind.

Experience in the refinery and laboratory experiments have demonstrated that an earth having the gravity of 0.176, or slightly over that number, is best suited for filter-press work. As soon as the gravity rises above this accepted standard, the efficiency of the earth falls. Since it is rather upon the volume of earth than upon the added weight that its defecating and filtering property depends, it follows that a heavy earth, *measured* into the liquor by volume will consume a greater quantity than a lighter brand. Nor will an equal volume of a heavy earth give as satisfactory press-filtration as one of lower gravity. But again, some earths of low gravity will be encountered that upon trial will exhibit limited and slow-filtering qualities. It will be noted that in such earths the diatoms are very much broken and therefore tend to form a compact layer on the filter-cloths and thus impede filtration.

If ten grams of earth in 100 cc. of water is well shaken and allowed to stand in a tube of 1-inch diameter, after 1 hour leaves the water milky, the earth probably contains an excess of broken diatoms.

Supplementing these simple physical tests, a microscopical examination will always be desirable. From time to time comparisons of the lots of earth should be made as regards the relative conditions of the diatoms.

Under the microscope a desirable earth will reveal little sand, clay or any material other than diatoms. Nor should the diatoms be too much broken, and it is a good feature if the species observed be mixtures of the discus and rod types.

3. **Hydrochloric and Sulphuric Acids.**—Either hydrochloric or sulphuric acid may be used in the preparation of phosphoric acid or superphosphates. Both of these acids when pure, are water-white liquids. Concentrated sulphuric acid (H_2SO_4) has an oily appearance, hence its popular name, Oil of Vitriol. Hydrochloric acid (HCl) is also known as muriatic acid.

For technical use the ordinary commercial acids will, of course, be bought under contract and specifications relative to their strength and quality. The commercial acids are generally of uniform strength, and from the determined specific gravity the quantity of true acid present can be ascertained by consulting acid tables to be found in most works on chemistry.

Liquid commercial HCl is a solution of the acid gas in water; is of a greenish yellow color, and customarily has a specific gravity of 1.398, or 18° Bé. equal to 28 per cent of true acid.

Sulphuric acid of commercial quality is a heavy, oily liquid, brownish yellow in color and when of full strength has a gravity of 1.84, or 66° Bé., and is termed 66-degree acid.

The color to be observed in both of these acids is chiefly due to the presence of iron salts, but this may be neglected if not extreme, and other impurities are not excessive.

Both acids are likely to contain, as metal impurities, lead, arsenic and calcium, beside the iron referred to. Lead and arsenic are particularly objectionable when the acids are to be used for phosphate preparations, which later enter into refining operations. Neither of these acids are ever free from these poisonous metals, and therefore, occasional determinations of the presence of the metals should be made. Distinct traces may, however, be tolerated since the percentage of the acids used on the raw sugars is so small that even a trace of the metals vanishes in the material operated upon. Indeed, under refining conditions, the insistence on the entire absence of lead and arsenic rests rather upon sentimental than on practical reasons, and it is a matter of doubt whether these two acids are ever free from lead and arsenic except when they have been prepared for laboratory use.

4. **Phosphoric Acid and Soluble Phosphates.**—As pointed out in paragraphs dealing with defecants (page 57), soluble calcium phosphates may be regarded as typical chemical defecants. The chemical action and function, as there explained, is obtained by adding superphosphate of lime—which has an acid reaction—to the liquid to be defecated and later neutralizing this acid phosphate with milk of lime. Insoluble tricalcium phosphate is thus produced and materially aids subsequent clarification in the bag-filters.

But phosphoric acid itself may be employed as well as any water-soluble phosphate. When, therefore, soluble phosphates, or the acid is bought, it is important to determine the amount of soluble phosphate the product may contain. Ordinary soluble phosphates and phosphoric acid vary to a marked degree in respect to the available soluble phosphoric radicle or the acid.

The strength of a product is therefore usually expressed in terms of *phosphoric anhydride,* or the water-soluble P_2O_5, which in combination with water forms phosphoric acid, thus:

$$3\ H_2O + P_2O_5 = 2\ H_3PO_4 \text{ (Phosphoric Acid).}$$

In order to show the identical results obtained—as far as defecation is concerned—by either the use of phosphoric acid or a water-soluble phosphate salt, comparison may be made as to the reactions between:

(a) Superphosphate of Calcium and Lime

$$CaH_4(PO_4)_2 + 2\ Ca(OH)_2 =$$

$$\overset{\text{Trical. phosphate}}{\overline{Ca_3(PO_4)_2}} + \overset{\text{Water}}{\overline{4\ H_2O}}.$$

(b) Phosphoric acid and Lime

$$2\ H_3PO_4 + 3\ Ca(OH)_2 =$$

$$Ca_3(PO_4)_2 + 6\ H_2O.$$

In either case insoluble tricalcium phosphate is formed, which is the main purpose of the induced reaction.

Many refineries, and especially those making, say, 15 to 20 per cent of soft sugars, have an ample supply of discard char and chardust from which to manufacture the required soluble phosphates.

Discard char and dust contain from 30 to 33 per cent P_2O_5, combined with calcium forming the basal constituent of bone. The so-combined P_2O_5 can readily be obtained either in the form of a soluble phosphate or as free phosphoric acid by digesting the ground char or dust with a calculated quantity of either hydrochloric or sulphuric acid.

However, phosphoric acid and superphosphates have now but a restricted use and are seldom employed as defecants where bag-filters have been discarded for the more efficient filter-press, and therefore, this brief discussion of the subject may suffice with the illustration of the reactions of lime with phosphoric acid and lime with superphosphates.

Claassen points out that acid phosphate yields one and one-half times as much precipitate as H_3PO_4, and is therefore preferable for the production of a precipitate in defecation.

$$\overset{\text{Acid phosphate}}{\overline{3\ CaH_4P_2O_8 + 6\ Ca(OH)_2}} = \overset{\text{Trical. phosphate}}{\overline{3\ Ca_3\ P_2O_8 + 12\ H_2O}}$$

$$4\ H_3PO_4 + 6\ Ca(OH)_2 = 2\ Ca_3P_2O_8 + 12\ H_2O.$$

5. Sulphur.—When sulphur is to be used as a bleaching agent for liquors from which soft sugars are to be boiled, a refined article should always be used. Refined sulphur is put upon the market as "roll sulphur" and sometimes as "block sulphur." In either form, if it has been produced by the sublimation of crude sulphur, the refined product will contain not more than 0.5 per cent of impurities, usually in the form of an "ash," and possibly a very small amount of water. The recently introduced Louisiana sulphur, obtained by the Fransch Process, is of a very high degree of purity; from 99.9 to 99.98 per cent sulphur.

Sulphuring Apparatus.—Sulphur is used in the form of a gas —sulphurous acid gas—and to produce this, the sulphur is burned in a sulphur-oven. The gas is passed through an absorbing apparatus against the stream of liquor it is required to bleach. A sulphur-oven and an absorbing tank or tub are the essentials for "sulphuring."

A simple and efficient sulphur-oven may be made of cast iron plates to form an oblong box, 36 inches x 12 inches x 6 inches. At the front and at the bottom of the box a horizontal slot is provided into which a cast iron tray may be slipped and must be adjustable for the admission of the air necessary for the combustion of the sulphur placed in the tray. At the back and at the top of the box a circular hole (6 to 8 inches in diameter) is made and connected by tyle pipe to the "sulphur-tub." Air is drawn through the oven and tub by means of a steam ejector.

The sulphur-tub should be made of wood and preferably square. Square tubs are more easily kept liquor and air tight than circular ones and should be about 5 to 6 feet high and 3 to 4 feet square. The tub is provided with perforated distributing plates or shelves. The liquor is run into the top of the tub, finds its way through the perforations against the ascending sulphur fumes. The descending liquor rapidly absorbs a sufficient quantity of the sulphur gas (SO_2) and flows from the tub into a receiving tank.

Diagrammatically the scheme may be shown thus:

Oven and sulphur-tub of dimensions given should have a capacity for the sulphuring of 600 cubic feet of liquor containing 22,000 to 25,000 pounds solid matter, to a maximum SO_2 content of 400 milligrams per liter. Further, it is advisable to build sulphuring systems of a capacity conforming to the capacity of the vacuum pans they are to serve. The system just described is suitable to a 12-foot pan.

Another type of sulphur-oven is used in cane and beet sugar factories, where larger streams of SO_2 gas are required. Such ovens are operated by compressed air forced over the ignited sulphur; the resulting gas, therefore is blown through the absorbing tank or tub.

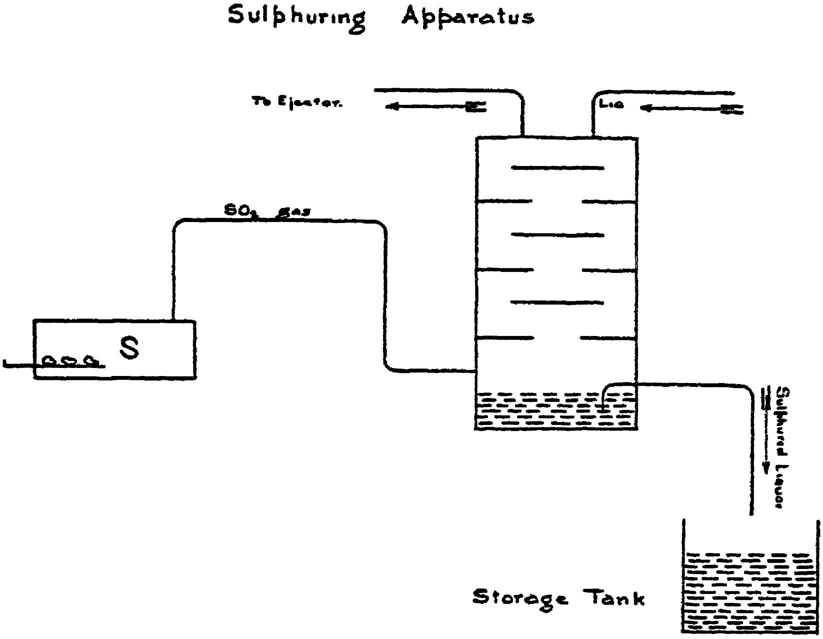


Fig. V.

The fear of the partial oxidation of the SO_2 gas into sulphuric acid in the presence of a moist atmosphere, seems, in the opinion of the writers, somewhat groundless, but an excess of air should be avoided. However, G. L. Spencer in "A Hand-book for Cane Sugar Manufacturers," writes, page 59 as follows:

"It is very essential that the sulphur-stove, no matter what its type, be provided perfectly dry, clean air for the combustion in order to avoid partial oxidation to sulphuric acid. An adaptation of the following arrangement for closed stoves should be used with the induced draft type.

"The drier is preferably a cast iron box provided with a tight-fitting door and trays for holding layers of quick lime. The air enters the box at the bottom and passes over the lime on its way to the compressor. It is advisable to place a settling-chamber between the drier and the air-compressor to catch the lime dust. A drier about 3 feet x 3 feet x 4 feet is usually large enough for a stove of 8 square feet fire area."

6. **Sodium Salts.**—Under this head may be included sodium carbonate (washing soda), sodium hydrate (caustic soda), sodium phosphate and sodium silicate (soluble glass).

The qualities of these salts will, of course, be determined by the chemist, and their applications to occasional adjunctive operations will be directed by him in most cases. None of these salts can be said to find use in the regular operations of a refinery. The particular functions, therefore, of these salts will appear when their use is discussed under adjunctive operations and control.

7. **Filter-cloths, Blankets and Bags.**—Standard fabrics are usually established by each refinery, and consequently checks upon delivery are necessary to ensure the maintenance of the quality required. Treatment and care of the fabrics will appear under later headings.

8. **Carbonic Acid Gas.**—This gas finds now only infrequent use, and, indeed, the one time "gassing" of char-filters by blowing boiler flue gases—containing 10 to 12 per cent CO_2—through a filled char-filter, has been discontinued. Should, however, a small quantity of this gas be required for the precipitation of excess lime in liquors, it may be found in the flue gases. If these be drawn from the chimney, passed through a gas-washing tub, to free them from sulphurous acid, the carbonic acid gas they contain may be used for the purpose indicated.

9. **Decolorizing Blacks (Amorphous, and Other than Char).**—During the last twenty years a great number of decolorizing blacks have been offered as forming the basis of new decolorizing methods for various purposes, but so far few, if any, have found acceptance as efficient and economic substitutes for char in the refinery. In some localities and under favorable circum-

stances a few blacks have gained a footing—whether permanent or not has yet to be determined—but as rivals to char-filtration not one of them has been successfully applied for any length of time.

The authors will, however, in later paragraphs discuss the possibility of using such blacks as auxiliary agents in connection with char-filtration.

For the present let it suffice, in this review of accessory materials, to say, that efforts directed towards the production of dusty, sooty, highly-comminuted decolorizing blacks has been very diverse. Generally speaking, vegetable—and sometimes animal—tissues have been mixed with lime, clay, diatomaceous earth, etc., and the mass subjected to distructive distillation somewhat along the lines employed in char production. The charred mass so obtained is variously treated and finally appears as a fine black powder, or, if desired, as agglomerated particles. A few blacks have been marked under fanciful proprietory names, such as "Carboraffin," "Molasacarb." etc., and offered at remarkably high prices. Carboraffin, for instance, was quoted at $350 per ton.

A fairly complete discussion of the manufacture, use, valuation, etc., of amorphous blacks will appear in the chapter dealing with the chemistry of decolorizing black and bone-black.

10. Bone-black or Char.—Of all the accessory materials used in the refinery, bone-black, animal-black or char, is predominantly the most important. Its preparation, handling, revivification and general use involves a considerable expenditure, the operation of much machinery and maintenance of extensive building. A large amount of fuel and many hundred cubic feet of boiling water are used to keep the "working chars" in condition and up to the required standard quality.

When the char supply of a refinery is maintained by purchase from a manufacturer every lot should be carefully analyzed and experimental filtrations made upon an average sample of 25 to 50 pounds.

If the char is to be manufactured from raw bones purchased by the refining company, and, therefore a bone-kiln operated, char may be cheaply and profitably produced under proper supervision. The purchase and selection of raw bones and the management of the kiln then also becomes a charge upon the superintendents, and some judgment is to be exercised in the handling of the different qualities of bones that may be available for char making.

Bones, then, become the raw material for char production, and must be dealt with in so far as their quality may contribute to the yield and quality of the char obtained from them.

11. Raw Bones; their Character and Quality.—The bones of the beef* and the horse are the best raw material; from them is obtained the greatest yield and the best quality of char. But all parts of the skeletal frame are not equally serviceable in char production, and some parts of the bones, particularly the knuckles and tissue immediately surrounding them, yield only an inferior char, while from the cartilaginous tissues a glistening, flaky black is produced that has no decolorizing power whatsoever.

Bones from smaller animals, such as the sheep and hog are inferior raw material, since the proportion of hard tissue is lower than that in the corresponding bones of the beef or the horse.

In order of their yield value bones may be classed as:

1. Femur and tibia, known as "shanks" or "marrows."
2. The pelvic and shoulder bones.
3. Skulls and jaws.
4. Ribs and vertebrae.
5. Knuckles (very inferior).

Again, another important factor relative to the valuation of bones generally, is their condition at the time of their delivery at the kiln. For convenience they may be cast into three classes:

(a) Green bones.
(b) Extracted bones.
(c) Mixed bones.

(a) Green bones may be more properly regarded as fresh or whole bones, *viz.*, such that have not been dried nor subjected to any extraction process for their fat and gelatine. Such bones may then contain all the constituents of the natural bone, with more or less adhering flesh, according to the care exercised in its removal. Fairly dry, clean green bones are good char producers in spite of the fact that bulk for bulk they may weigh 10 to 15 per cent more than extracted bones.

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*Beef bones are superior to horse bones.

(b) Extracted bones are those from which fat or gelatine, or both, have been removed either by (a) steaming for gelatine or (b) chemical extraction for their fat by a solvent such as benzine. If these extractions have not gone beyond 50 to 60 per cent of the fat and gelatine of the whole bone, such extracted bones may be regarded as still able to yield a char of required chemical composition and sufficiently hard to withstand attrition during use. But bones from which all, or nearly all fat and gelatine has been extracted, will produce char deficient in carbonaceous matter and of a brittle nature.

(c) Mixed bones can be valued only in so far as a careful inspection may indicate the proportion of the classes (a) and (b) in a lot, and also with regard to the relative quantities of shanks, skulls and so forth.

A fourth and quite distinct class of raw bones has, however, within recent years, been placed on the market, and has become known as crushed bone. These bone have evidently been carefully cleaned, dried and crushed in such a manner that they are not subject to putrefaction and can be transported and stored without offence. Crushed bones are offered in sizes ranging from a coarse meal to a large bean. As the siftings are uniform, the larger sizes are well adapted to retort work and the yield is frequently 40 per cent higher in char than the best of the (a) and (b) class. The data relative to their handling for char manufacture will therefore also be considered among the other classes just mentioned.

DESTRUCTIVE DISTILLATION OF BONES

In establishments that operate on large quantities of bones and aim at the utilization of all products obtainable from them, destructive distillation is preceded by the extraction of grease and gelatine as mentioned in antecedent paragraphs. In refinery bone-kilns, however, having the manufacture of char solely in view, the extraction operation is generally dispensed with and bones of the three classes are charged into the retorts in mixtures determined by the stock of the grades on hand and with the aim of securing uniform kiln operation.

When the bones, then, are to be carbonized they are shoveled into a battery of retorts built and walled in such a manner that they can be raised to a cherry-red heat (about 800° C.) by the combustion of coal on grate G, shown in diagram subjoined. In this sketch are shown the essential features of a bone kiln. In this R is the retort, about 10 feet long and D-shaped in cross-section (18 inches x 14 inches). C is a cylindrical condenser, half filled with water into which vapor pipes (vp) dip below the surface of the water which catches the condensable vapors but allows, under prevailing temperature, the non-condensable gases to pass out through gp into the combustion space of the kiln there to be burned, contributing by their heat of combustion to the economy of the fuel. Similarly, the pipes op deliver the condensed "Bone-oil," O, to the furnace where it is likewise consumed. The arrangement of a three-battery retort kiln is shown in the front view of the sketch. Sometimes five or even seven retorts constitute a battery.

Operating a Bone Kiln.—As the battery of retorts and the brick work immediately around it must first have attained the temperature required for the carbonization of the bones, the first charge of bones cannot be attempted until after the fire has been heating the apparatus for 3 to 4 days. As soon as the interior of the kiln has reached a clear red heat, about 250 pounds of bones is packed into each retort of the battery. The end plate, P, is quickly fastened into place and tightly clamped against the face of the retort, thus closing all access of air from the bones. The organic matter on and in the tissues of the bones at once begins to decompose forming a great variety of more or less volatile compounds and a residual amount of carbon which slowly deposits itself onto the mineral tissue of the bone.

It is, of course, entirely beyond the scope of this work to bring these distilled substances into review, but it may be well to point out broadly the nature of their origin. The organic matter of bone tissue contains the elements, hydrogen, oxygen, nitrogen, sulphur and phosphorus. In the process of decomposition of organic matter these elements regroup themselves, into (a) gases, (b) water, (c) ammoniacal liquids and (d) bone-oil.

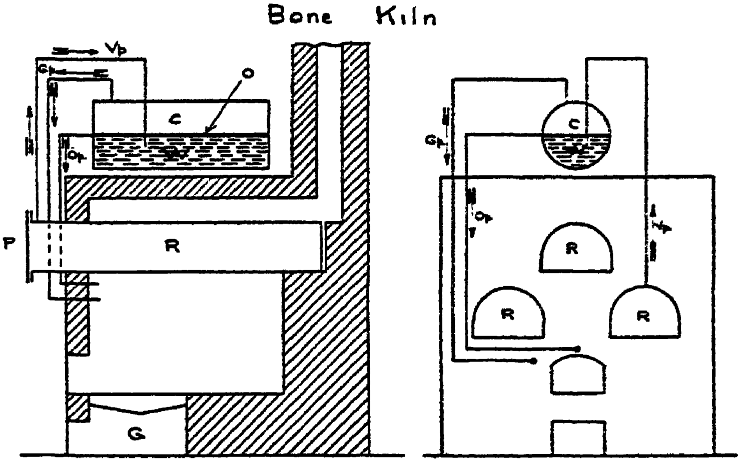


Fig. VI.

The gases bubble through the water, W; pass along gp and are burned over the fire. Water and ammoniacal liquor are condensed in c and eventually run to waste. Bone-oil gathers on the surface of the water in the condenser, flows down op and is ignited in the neighborhood of G.

The only profitable manner of conducting bone-kiln operations is to keep the carbonizing process going on under as uniform conditions as possible, so that the temperature of the kiln shall vary but slightly and the raw bones charged and the charred bones be withdrawn at regular intervals. Intermittent work not only involves waste of fuel, but also undue deterioration of kiln material. A constantly maintained heat is not so injurious to retorts and brick arches as unnecessary cooling and reheating. With changes of temperature expansion and contraction tends to loosen masonry joints and may even cause the retorts to crack or, at least, warp and thus shorten their life.

When the kiln has attained its necessary temperature and the first charge made, the fire is regulated so that together with the burning coal and the combustible distillates, the heat supply shall be maintained during the entire period of the "burning." With proper care, well-built brick work and sound retorts an uninterrupted burning may be kept up from 4 to 6 months—even 8 months of continuous operation has been reached before signs of weakening are noticeable and the kilns have to be cooled, and repairs made.

With a fairly uniform stock of bones on hand and arriving from time to time, the period of the charges can be fixed; 6 hours will usually be found adequate. Between charging hours preparations are made to handle the red-hot carbonized bones. For each retort in the battery a canister is provided This canister is made of wrought iron, is a cylindrical vessel (36 inches x 24 inches) with a tightly fitting cover, and of a capacity to just hold the volume of the charred bones as they are drawn from one retort. At the proper time the canister is put upon a convenient truck and placed in front of the kiln and under the mouth of the retort first to be drawn. Plate P is removed and the carbonized bones raked, as rapidly as possible, into the canister and the cover *immediately* put on. During the drawing the hot bones may burst into flame, therefore, it is desirable that the work be done as rapidly as can be so that a minimum of carbon shall be lost. The discharged retort is immediately recharged with a new lot of raw bones, closed with the plate P and carbonization goes on as before.

As any access of air to the hot-burned bones will reduce their carbon content, the cover of the canister is luted, for further protection from the air, with a mixture of sand and clay and in this condition set aside to cool. Twenty-four hours will be ample for this and the charred bones may then be taken out and are ready for grinding.

Character of Burned Bones.—If the "cooking" has been ample, the charred bones should have a uniform, dull-black appearance, and if a shank be broken, the inner tissues should never show a brownish-black ring. The presence of this ring will indicate insufficient burning. At times some of the bones may have a greyish film upon them, due to local decarbonization experienced during the drawing. If this film is not deep or excessive, the condition may be disregarded. Best judgment, however, may be passed upon a particular burning by noting the smell of a freshly opened canister. Properly burned bones may have—and usually do have—a distinct ammoniacal odor, and should never give a faint odor of burned hair, or, indeed, any pungent smell suggestive of the incomplete destruction of the nitrogenous organic matter of the raw bones.

Overburned or overcooked bones are more difficult to detect, and such defect would have to be excessive indeed before any physical indications of fused bone tissue could be observed. Chemical analysis only might aid in fixing the fact. On the whole, however, overburning is hardly ever experienced.

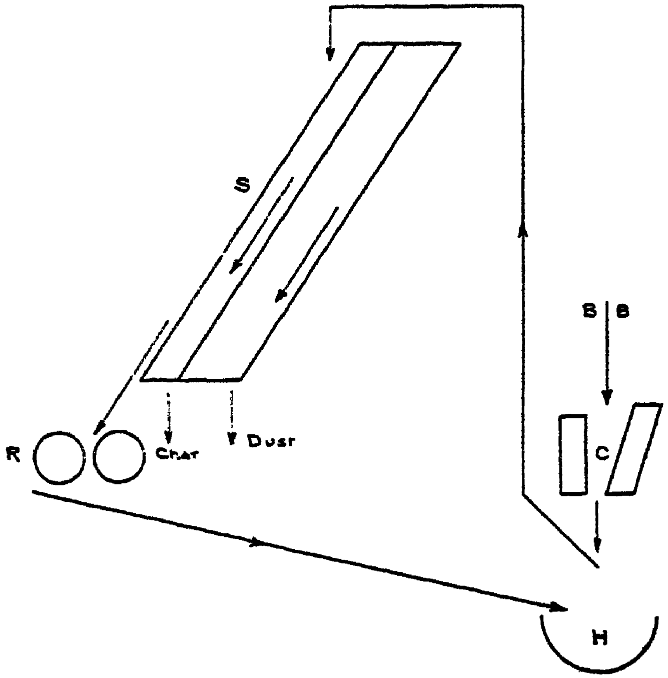
Grinding and Screening of Burned Bones.—The care in grinding and screening of burned bones should be directed to obtain the maximum production of char grains of the standard size fixed upon by the refinery requirements.

The reason for selecting a certain size of grain will appear later, in the meanwhile suffice it to say that the chars offered to sugar refineries are usually 16 to 30-mesh chars. This means that the char delivered will contain grains passing through a 16-mesh screen but will pass over a 30-mesh. The opening in such screens will be respectively 0.04 square inch and 0.02 square inch.

When a refinery makes its own char, however, it is advisable to sift the ground bones to a range of 10 to 50-mesh, thus having grains varying from 0.05 inch to 0.01 inch; the reason for this will become apparent when the chemistry of char comes into discussion.

The chief features of grinding operations may readily be followed by the aid of the accompanying sketch (Fig. VII) in which C is the jaw-crusher through which the whole burned bones (BB) are passed.

From C the crushed bones fall into the hopper, H, and are carried by an elevator—shown by arrowed lines—to a Newago screen, S. The screen separates the crushings from any bone that has been broken to char size and also delivers any dust to the dust bag. The large pieces pass from S through the rollers, R, and slide back into H and are again passed over S, producing char and dust.



Arrangement of Char Grinding Apparatus.

Fig. VII.

The preliminary crushing of burned bones has been shown to be of considerable advantage. When a crusher is not used and R is placed in the room of C, much more dust is produced consequent upon the rasping action of the rollers upon the larger pieces of bones "riding" the rollers with the formation of dust. In the jaw-crusher on the other hand, the pieces are instantly broken to a size that will immediately be gripped by the rollers and ground to char.

The subjected Table IX and Diagram IX will show the varying yield, experimentally determined, of char from five classes of raw bones, in which:

M. Represents 155 tons of mixed bones.

G. Represents 24 tons green bones, horse and beef bones, containing much flesh and fat.

S. Represents 13 tons of skulls; very dry.

K. Represents 25 tons knuckles; partially extracted. Poor char.

C. Represents 110 tons "crushed" bones. Excellent yield and good char.

TABLE IX

Per cent yield	M	G	S	K	C
Of BB on RB	47.7	62.0	65.8	71.0	69.3
Of char on RB	37.3	50.6	51.6	55.2	55.6
Of dust on RB	10.2	11.4	14.1	16.0	13.7
Per cent coal used on RB					
B. t. u. of coal 13,000	31.0	28.4	42.7	28.2	36.4

BB = Burned Bones; RB = Raw Bones.

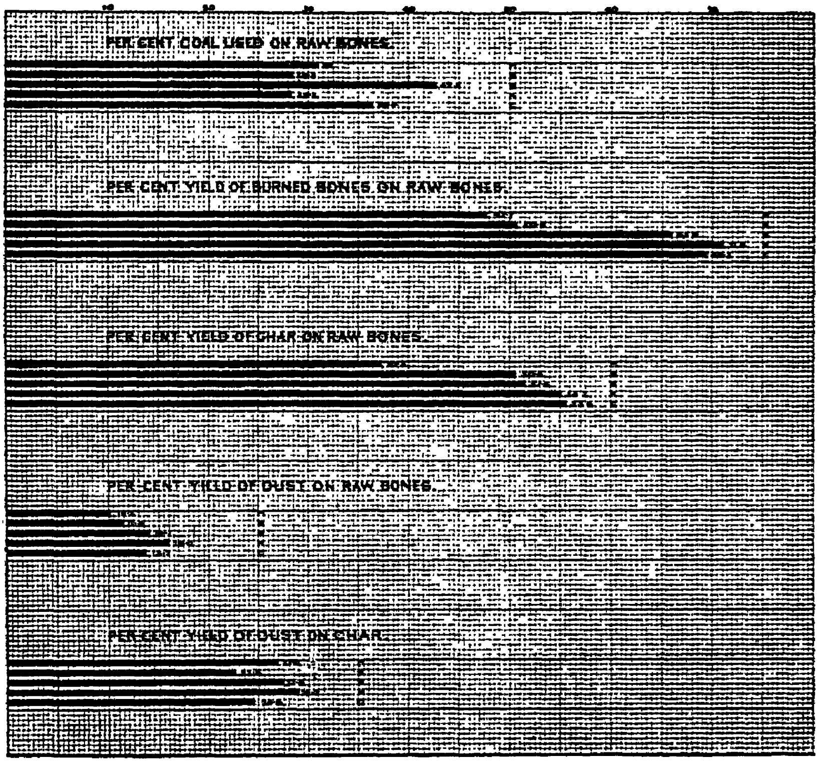


Diagram IX.

From a critical analysis of these data a fair estimate of the relative cost of char produced may be obtained, but it is necessary to add that all the factors should be considered as each yields its quota to the final result. For instance, low-coal consumption is characteristic of classes M, G and K, while it is higher for S and C. Again the yield of char is high for K, G, and C. In this respect it must be remembered, however, that K char is very inferior, having something like 20 per cent less decolorizing power than G or C, and probably other inferior qualities relative to adsorption of ash, etc.

The high-coal consumption by the burning of S and the low consumption by K, may be attributed to the absence of fat and flesh in S and to the presence of much cartilaginous matter in K, which gives off combustible gas and distillates.

Finally the cost of char production may be said to approximate to two and a half times the original cost of the raw bones.

BONE-BLACK OR CHAR

As regards char as an accessory material it is necessary in this connection to point out what features of a purchased char should receive attention, and the quality of the char secured by contract stipulations.

These stipulations should relate chiefly to comminution of the char; its percentage of dust, *viz.*, the maximum allowed char grains smaller than those retained by the finer mesh.

As char is somewhat hygroscopic, the maximum amount of moisture should be stipulated and the method of water determination also indicated by which the moisture is to be established, so that no dispute may arise by disagreements in this respect.

Chemical analysis should, of course, always be made and filtration on laboratory scale is advisable. For this purpose a 10 to 20-pound sample is necessary.

In other respects char-handling, its care and preservation will be dealt with in a chapter exclusively devoted to this important accessory.

CHAPTER II

ADJUNCTIVE OPERATIONS

Along with the main operations of a refinery there are a number of side activities, the object of which is the keeping of apparatus in good working condition, and the reintroduction of sugar liquors into the process from which these have been temporarily diverted. Of course under adjunctive operations repairs and mechanical attendance are not to be considered and these are beyond the scope of this work. But there is the attention that should be given apparatus, that working in cycles, need regular and periodic care to keep them up to their maximum capacity. Again, from heavy sugar liquors passing through filter-cloths and bags there accumulates a deposit of lime salts which must be removed from time to time. In press and filter work there is a periodic washing of presses and the recovery of sugar from the filter-aids used. This will necessitate the establishment of a system fitted into the general working of the house. (See Diagram, p. 159).

For convenience, adjunctive operations may be divided into five classes, *viz.*:

I. The recovery of (a) sugar from fabrics and (b) the care of these fabrics.

II. The recovery of (a) sugar from "mud cakes;" (b) regeneration of filter-aids.

III. Concentration of Sweet-waters, etc.

IV. Cleaning of Pans and Effects.

V. Care and Management of Working Chars.

I. FABRICS

(a) When the raw sugar has been emptied from the bags, now almost the exclusive containers of raw material, sugar grains naturally cling to the fabric, and if the sugar should accidently have been damaged by water a certain amount of molasses will also have been absorbed by the container. If a centrifugal sugar contains an unusual amount of moisture—molasses—the grains will adhere tenaceouusly to the fabric. Some molasses and muscovado sugars during storage, and particularly when they have been subjected to high piling, will have some of their moisture in the form of molasses forced into the tissues of the bag, so that in such cases 2 and 3 pounds of sugar and molasses may remain in and on the bag.

Good, clean bags, as dry centrifugal sugar containers, may be brushed free from adhering sugar, and this brushing is occasionally practiced and found satisfactory. In most instances, however, washing or steaming will be the most economic and satisfactory method. In some refineries brushing and washing are practiced in combination.

A properly washed sugar bag will not only have practically all the sugar washed out, but any adhering foreign particles will also have been removed and the eventually dried bag will be serviceable for a variety of purposes.

Although the washing process is of the simplest and does not vary in principle, local conditions are responsible for a great diversity of details in the actual manipulations.

Probably the most generally accepted method is to give the bags a series of steepings in hot water by passing them from one rectilinear tub to another against a stream of hot water. Given, say, three tubs of capacity to meet the demand, the water in the last tub will be kept about 1° Brix, that in the second about 3 to 5, while in the first tub the density of the water may be allowed to reach 7 degrees.

As the bags are moved from tub III they are passed through a wringer (W in the sketch) and may be carried by a conveyor to the drying department or otherwise disposed of.

As will appear in Chapter III, Control, the wringing is worth the outlay in several ways. First, the wet bag will contain much less water and a minimum amount of sugar when so treated. Secondly, much less heat will be required in the drying.

The seven-degree sweet-water is pumped away to a common sweet-water tank from which it is sent to the effects for concentration, or it may be used tc dilute the heavy raw syrups in the treating tanks. If the raw syrup is to be boiled for remelt, the sweet-water may also be sent to the suction tank of the remelt pan to reduce the charges as required.

Although raw sugar bag washings may be derived exclusively from bags that had contained centrifugal sugar of 97 purity, the sweet-water should not be used for melting affinated sugars. During the steeping and washing an appreciable amount of color is removed from the jute fiber and, no doubt, other soluble substances also, so that the purity of the sweet-water from such bags may fall to something like 80 purity, and, of course, should rather be used to enrich raw syrups than reduce the 99 purity affinates. In fact, as regards color, this sweet-water has been shown to carry three to four times more color than the raw sugar itself.

Inasmuch as the residual sugar in filter-press blankets and in bag-filter bags is removed coincident with the mud upon them, the operation will receive attention under Section II.

(b) Fabrics through which sugar solutions are being continually strained, accumulate a layer of calcium salts, organic and inorganic which simple washing will not remove. As these salts deposit themselves upon the cloth they not only form a tenaceous, impenetrable skin upon the fabric but also invade the tissues and interstices of the mesh, so that the filtering capacity of bag or filter-cloth gradually falls from its original efficiency and eventually must be cleaned.

Sodium carbonate—washing soda—is the best agent for this purpose, and as it may be used several times, it is advisable to have a store of this salt in solution ready for use when required. A solution of the anhydrous salt is made up to 5° Brix, equal to a 3 per cent solution.

In the case of the Sweetland press-cloths, the fabric may be cleaned *in situ*. After the press has been put out of commission for this purpose and the cloths have had a flushing with hot water to remove the last traces of sugar, the press is filled with the hot (190° F.) soda solution. Ten to 15 minutes is allowed for soaking, after which the soda may be run off, used on a second press or sent back to the storage tank. Three successive charges of hot water are now sent through the press, allowing 3 to 5 minutes for the water to extract the residual soda. It will be found that the third charge of hot water will be almost free from soda; indeed, a small residue of soda in the cloth, indicated by a slight alkalinity, is not at all detrimental and may be allowed to remain —the first flush of the next inflowing liquor will carry it off.

Cloths from plate and frame presses and bag-filter bags may be steeped in the same hot soda solution and rinsed in successive lots of hot water until free from soda. Scrubbing the cloths after the washing with a medium hard brush will further open the fabric and improves its filtering qualities.

By such soda treatment bags and cloths can be restored to within 80 per cent of their original efficiency and used until the fabric becomes too weak, when they must be discarded.

II. RECOVERY OF SUGAR FROM MUD CAKES

(a) One-fifth of 1 per cent may be accepted as the mean amount of insoluble matter in raw sugars entering a refinery, to which must be added a further 0.3 per cent of filter-aid material. If bag-filters are also employed for clarification of raw syrups, one-tenth to one-fifth of 1 per cent of tricalcium phosphate will be formed in the sugar during treatment. The total weight of solids to be handled during the refining of, say 1,000,000 pounds of raw, will be about 6,000 pounds, and as this, in the form of "mud," will be discharged from the presses as a "cake" containing from 40 to 70 per cent of water, about 10,000 pounds of the mud will have to be carefully washed so that a minimum of sugar be lost.

Press and bag-filter work produce a quantity of solid residues characteristic of each.

Mud from the presses in no way resembles the mud from the bag-filters. Press mud, indeed, is nothing more than the diatomaceous earth mixed with a small quantity of fiber and suspended matter that has persisted in hanging to the crystals of the affinated sugar. Bag-filter mud, on the other hand, will vary somewhat according to the nature of the main constituents of the suspended matter of the raw sugar. The argillaceous matter and finer sand particles will find their way into the raw syrups, and these together with the tricalcium phosphate of the treatment will accumulate in the bags and eventually be discharged also as cake, but one rather more tenaceous and not so readily freed from its absorbed sugar.

The muds must, therefore, be kept separate and treated differently in the final washing process.

REGENERATION OF MUD

(b) A proper mud pressing followed by a thorough sweetening off in the Johnson plate and frame press will result in the forming of a firm "cake" between the plates of the press. After cooling with a stream of cold water and upon discharging the press the mud will fall from the plates in a sufficiently dry state to be readily handled. As has been intimated, this mud is practically the original diatomaceous earth plus a small amount of suspended matter and some fiber from the sugar bags. The diatoms have in no way been injured and can be seen intact, or no more broken than those of the fresh, unused earth. We have, in fact, in this wet mud a substance that can be restored to within 80 per cent of its original worth by drying, calcining and grinding. The cost of regeneration, or rather the profitable regeneration, will, of course, depend upon the relative price of coal and earth. Probably the best way to regenerate the earth will be to carry on the operation in an adapted lime kiln.

It has been shown however, that the wet mud can be regenerated in raw-bone retorts with access of air, in about 12 hours' burning and that the resulting baked lumps can be easily broken and sifted to a fairly uniform powder. The problem of the most efficient method of regeneration is still the subject of experimentation.

(c) Washed mud cakes from syrup presses, however, cannot be regenerated as they are heavily charged with sand and clay, and may also contain an appreciable amount of tricalcium phosphate when they originate from bag-filter washings.

In that event it is necessary to establish certain conditions so that the subsequent mud pressing in the Johnson presses shall proceed expeditiously and with the minimum production of sweet-water.

The two essential features are, (1) density and (2) alkalinity of the mud going to the presses.

(1) The density of the mud sweet-water should never be allowed to rise above 7° Brix, otherwise too much hot water will be required for sweetening-off, and the viscosity of the mud cake between the press plates will tend to the formation of slushy cakes from which it will be difficult to wash the adhering sugar. If the mud sweet-water density should be found to be above the prescribed figure, it can be reduced by adding thin sweet-water.

(2) Mud of acid reaction, or even neutral reaction will press-filter very poorly. Not only does the pressed sweet-water persistently run "cloudy," but the press will clog before the interplate spaces have been filled with mud. This condition would be followed by excessive sweetening-off water and poor press work generally.

A generous addition of milk of lime corrects all this. Indeed, the muds should be limed to an alkalinity of say, 1,000° at least. Vide, Chemical Tests of Liquor Reactions, page 170.

III. CONCENTRATION OF SWEET-WATERS

The principles and practice of multiple effect evaporation has received attention in Part II, Chapter IV, page 106.

In so far as concentration of sweet-water is to be regarded as an adjunctive operation, its aim is simply to bring all sugar solutions below a certain density up to the standard required for char-filtration. Not infrequently throughout this work reference has been made to the large volumes of sweet-water that must be expeditiously handled in order that no blockage be caused by their accumulation.

In this connection a suggestive method for the disposal of sweet-water may appropriately find place here. The accompanying Diagram X will illustrate a principle, rather a fixed plan, but also shows how sweet-waters are produced as well as the disposal of them.

In this diagram Sweetland presses are shown as clarifiers for syrups as well as affination liquors. Recent advances in the use of Sweetlands as efficient presses for raw syrup (Greens) has justified this arrangement.

In the Diagram:
SL Sweetlands for raw liquor.
SS Sweetlands for raw syrups.
JL Johnson presses for mud from SL.
JS Johnson presses for mud from SS.
HW Hot water supply.
G Gutters.
E Effects.
P Vacuum pan.
p Pumps.
I Heavy sweet-waters containing mud passing through JS or JL.
II Light sweet-waters containing a small amount of mud.

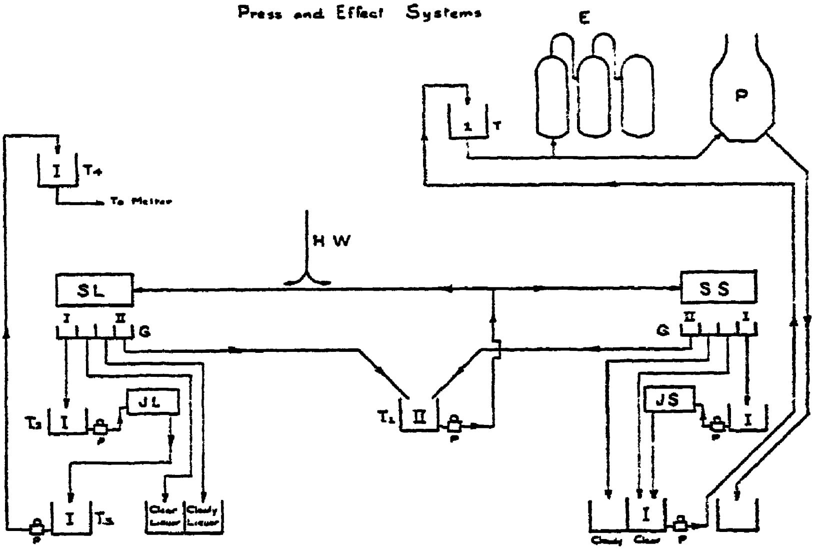


Diagram X.

Beginning, then, at the stage when sweetening off of, say, the SL must be undertaken, this press would at the time be filled with unfiltered liquor, and the leaves, seventy-two of them, covered with mud. The unfiltered liquor, remaining in the press, would be run off and sent back to the treating tanks. On the leaves of the press would remain from 100 to 200 pounds of syrupy mud, containing something like 20 to 25 per cent of sugar solids.

Under the method to be described, the sweet mud on the leaves would be removed by two operations with eventual recovery of the sugar in the mud, finally discharged from a Johnson press, in the form of a "cake."

Longitudinally placed within the press and over the leaves, there is provided a "sluicing pipe" with small holes so arranged that a stream of hot water—or sweet-water II from T1—can be directed against the mud. This so directed stream washes the sweet mud to the bottom of the press from whence it flows, by way of the gutter I to T2. This is the first sluicing, and produces a muddy water, I, having a density of from 5 to 7 Br., according to the volume it may be desired to produce, or as the nature of the mud may necessitate. From T2 it is pumped through JL and run into T3. From JL the sweet-waters issue as clear solution, free from all suspended matter and earth, and of a purity (92 to 93) to fit as a solvent of affinated sugar, or if the color be too high, it may be used as a base for boiling raw remelts. From T3 it is pumped to T4.

This preliminary sluicing, however, does not completely remove the mud from the leaves, therefore after a sufficient quantity of sweet-water, I, has been produced, a second sluicing is begun and these sluicings directed to gutter II, and from thence to T1, where they are stored until another press has to be sweetened off and washed. From there it enters the process again as sluicing water I and issues from the SL as described in the preceding paragraphs. Of course, the volume of sweet-water taken out of the system by way of T2 to T4 is made up by finally following the sluicing from T1 with hot water from HW thus maintaining a regular supply for the necessary work from day to day.

The mud derived from SL and gathered on the plates of JL must contain from 3 to 4 per cent of sugar, since it has been gathered from sluicing water I. This residual sugar in JL is recovered by flushing the JL press with hot water until the sugar has been washed out to such an extent that the washed mud does not contain more than 0.5, or at most 1 per cent of sugar on the wet mud. The exceedingly thin sweet-water so obtained is run into T1. Connections not shown on diagram.

As regards the sweetening off of SS a quite similar method is followed as can be seen by the indications of the diagram. In the pressing of raw syrups, however, and as recently developed, all lower purity sweet-waters are run together with the heavy raw syrup from the affination work, with the result that a low density raw syrup is obtained, varying from 43 to 45 Br. At this density it is possible to successfully pass the syrups through a Sweetland press, and to secure a filtrate sufficiently clear, so that after concentration, it can be sent to the char filters. If desired the light syrup (43 to 45 Br.) may be sent to the effects and concentrated to 52 Br. before pressing.

This method is obviously of great advantage. Bag-filter work always involves much labor in preparing the filters for each cycle, and also for washing the bags, etc. Press work on the other hand, requires little handling of material as is shown in the treatment of sweet-waters and mud in the preceding paragraphs. Furthermore, tricalcium phosphate is not necessary in press work; a prescribed amount of earth achieves clarification.

The one essential feature to successful pressing is that the syrups be sent to the presses under a slight alkaline reaction—so slight, indeed, that it does not interfere with subsequent treatment of the syrup.

IV. CLEANING OF PANS AND EFFECTS

Intimately connected with sweet-water concentration in effects and with the operation of the vacuum pans is the problem of keeping the evaporating surfaces of the apparatus free from "Scale." This so-called scale is a deposit of lime salts, usually a varying mixture of sulphates, sulphites and phosphates of calcium. Not infrequently a small percentage of carbonates may also be present. With the exception of sulphates, the other calcium salts should not be difficult to remove, but the scale, whatsoever its composition may be, has been "baked" upon the heating coils of the pan or the calandria surface of an effect, and it is no doubt due to the physical condition of the scale rather than to its chemical composition that difficulty is often experienced in its removal. Solvents that will decompose the powdered cake rapidly will react very slowly upon the scale itself.

Thin films of scale may, however, be dealt with without much loss of time and material, but as soon as such a scale attains any appreciable thickness it is difficult to remove. It is advisable, then, to set definite periods for cleaning effects and pans so that practically no film formation or scale really occurs.

The cleaning is best done by flooding coils or calandria with a weak hydrochloric acid water. To every 100 cubic feet of water necessary, 50 pounds of commercial, 18° Bé. acid is added. The apparatus, under partial vacuum—say 10 inches—may be filled with water to a height to cover the parts upon which scale formation is likely to take place, and then drawn in the calculated amount of acid through a lead pipe provided for that purpose. Steam is next turned on and the acid water allowed to boil at a temperature of about 190° F. for a few minutes. The hot acid water may then be permitted to stand at rest in the vessel for 2 or 3 hours. At the expiration of that time the acid water is run to the sewer; the man-hole of the pan or effect is opened and the interior of the apparatus washed with water delivered from a hose.

If there has been no undue accumulation of scale on the coils, a periodic application of this procedure will keep the boiling surfaces clean almost indefinitely. On the other hand, as soon as true scale has once formed, more troublesome methods may have to be resorted to.

Some authorities recommend a weak caustic-soda solution for this purpose, but this tends to attack the joints of the apparatus and cause annoying leaks. Washing soda, however, may be used as a preliminary agent, followed, after several hours of soaking, by the acid solution as just described. A 3 per cent washing-soda solution has a softening effect on the scale and expedites the action of the acid. When, however, the scale is very hard or thick, a 15° Bx. washing soda solution may be used before the acid treatment.

A heroic method for badly scaled pans consists of flooding the scaled coils with a 5 to 10 per cent solution of soluble glass (silicate of soda); heating for awhile and finally running the soda solution away while steam is left on the coils. Some of the soluble glass bakes on to the coils, and while the coils are still hot a stream of *cold* water is directed upon them. The effect is a violent contraction of the metal with a consequent splitting off of the scale. This method is effective, but should not be used except as a last resource.

Of course, scrubbing the coils with a soft iron brush is a laborious and long-drawn job, and should never be necessary if proper attention has been given to the pan relative to scale formation.

The presence of scale in pan or effect asserts itself in a distinct manner. Scale is a very poor heat conductor, so that the efficiency of the apparatus falls immediately upon only a slight scale formation. Again, in vacuum pans the splitting off of any scale during the boiling, will cause black specks to appear in the sugar spun from such a strike.

For these reasons, if for no other, scale formation should be systematically guarded against.

V. CARE AND MANAGEMENT OF WORKING CHARS

The care and management of working chars, as well as the entire char problem of a refinery are so intimately interwoven with the several stations of the house, that they cannot to advantage be practically discussed in any one respect and, therefore, here, as in preceding references to char, the whole matter is to be gathered into a succeeding chapter.

CHAPTER III

CONTROL

GENERAL

From the view point of control, the migration and rearrangement of the raw material to the refinery products is one of quantitative readjustment and the accounting for all intermediate steps as well as the balancing of the output with that of the intake of the technical departments. To achieve this, a system of technical book-keeping and accounting must be inaugurated and maintained by a staff qualified for the performance of the necessary observations and determinations upon which the balance may be cast. In order that this balance may not only be correct but likewise indicate wherein proper work has been done, or where improvements may be made or losses avoided, analyses and tests must be constantly made upon raw and refined material in all stations of the refinery. Records and data should be complete and comprehensive so that upon periodic tabulation a mental picture shall be obtained beside the mathematical array of the balance sheet.

Pursuant, then, of these ideas, control will be found to divide itself into five more or less interrelated investigations, all essential to a satisfactory estimate of a period of operation.

Control may be ranged under:

I. Analysis of raw material, refined products and residual products.

II. Tracing the material in process and keeping them under prescribed conditions.

III. Accounting for unavoidable losses incidental to operation and guarding against avoidable losses.

IV. Effective and economic use of accessory materials and the conservation of their function and quality.

V. Tabulation and calculation from data of the balance sheet.

I. As regards the analyses of all sugar products as well as the accessory materials, the chief chemist will, of course, be entirely responsible, and the superintendent staff can be occupied with this department only in so far that the laboratory forces shall prove themselves efficient and accurate in the tasks of this department. Where cooperation, however, is to be especially sought, between the house superintendents and chief chemist, is in the matter of securing the necessary samples for analysis. These must be taken, preserved and in every way guarded as prescribed by the chemist, and all details observed as may be arranged and provided for in every respect.

The most carefully executed analyses are valueless if the samples upon which they have been made are not truly representative.

To suggest methods of sampling and what particular samples shall in all cases be taken, would be entirely futile, since necessary analyses and tests may vary from time to time even during a period of operation, and would, of course, differ widely in each refinery. Suffice it to say, that during the course of this chapter the question of samples and method of sampling will naturally appear and then receive adequate attention.

Results and interpretation of analyses will likewise find application in their appropriate place.

Finally, in Part I, and generally throughout this work, there will be found ample suggestion as to when samples should be taken and analyses made. In regard to methods of examination, hand-books on sugar analysis and the like may be consulted.

II. Qualitative and quantitative data have to be regularly gathered with the view of keeping all materials in the various stages of refining in the condition prescribed, and to account, as perfectly as possible, for their quantitative migration through the successive stations of the house.

Of course, the obvious advisability of promptly gathering up any spilt material need hardly be mentioned, but it is always well to have someone appointed in the different departments who shall have this under his care. Beginning, then, with the raw sugar in the house, the first point at which loss of sugar can occur, will be in the washing of the raw sugar bags. The method of sugar recovery from these has been described, and therefore under this head it is but necessary to review a method for checking the unavoidable loss and keeping it down to the recognized figure.

Control at this station may be established by keeping the density of the last wash-water down to 1° Brix, and by the proper pressure being maintained between the rollers of the wringer. When these two factors are right little loss will be experienced and such as does occur can be checked in the following manner:

As the bags pass through the rollers, drippings are caught from time to time of the sweet-water from the bags as they are being wrung. The so-gathered samples of approximately uniform volume are poured into a common vessel, and if the analysis is to be made but once in 12 or 24 hours, a few grains of corrosive sublimate are dropped into the sample bottle to prevent fermentation or inversion of the sampled sweet-water. The polarization of the average sample is taken and noted.

Twenty or twenty-five of the wet bags are weighed and the weight multiplied by 5 or 4 as the case may be, thus establishing the wet weight of one hundred bags. Usually the dry weight of the bags is known beforehand, but it is advisable to weigh the bags after drying. From these data the daily loss of sugar remaining in the raw sugar bags can readily be calculated as follows:

$$\frac{\text{Lbs. of water in 100 bags} \times \text{Pol.} \times \text{No. of bags washed}}{100 \times 100} = \text{Pounds sugar lost.}$$

Example:

100 wet bags weight	420 lbs.
100 dry bags weigh	220 lbs.
Lbs. water in 100 bags	200 lbs.
Polarization of bag drippings	0.80
Number of bags washed	4,000

Then:

$$\frac{200 \times 0.8 \times 4{,}000}{10{,}000} = 64 \text{ pounds sugar lost.}$$

The reaction of the sweet-water in the wash tubs requires little or no attention except when any delay is experienced in pumping it away for concentration. In such a case a little milk of lime should be added to a slight alkaline reaction.

Attention has already been drawn to the magma syrup stored for affination work. The proper temperature is to be maintained and any tendency to acidity corrected. Magma density and affination work in centrifugals, etc., has also been disposed of, so that attention may now be given exclusively to methods of keeping reactions and treatments of liquors and syrups under observation. Densities and final temperatures, of course, are very infrequently changed—55° Brix at 180° F. for liquors; 53° Brix at the same temperature for syrups. Degree of alkalinity, however, may from time to time require modification, in accordance with the raw sugars entering the house.

Assuming, for the moment, that no other filter-aid is to be added than earth, it will be found that the melts from the affinated sugar are always slightly acid. This acid must be neutralized by milk of lime and, further, a determined alkalinity produced in the liquor for reasons which will appear later. This final alkalinity is determined by a prescribed method given to the control chemist, who makes daily reports or immediately draws attention to the administration should any irregularity be observed by him.

The quantity of filter-aid to be used in a batch of liquor is prescribed by the superintendent or the chemist, and regulated according to the pressing qualities of the raw material. About one-third of 1 per cent of earth on sugar solids is the normal quantity to be used.

It will also be the duty of the control chemist to see that the milk of lime be kept at proper density and the amount of filter-aid be regularly and economically applied.

TEST PAPERS AND INDICATORS

Referring once more to the control of liquor reaction, it is not only at the treating tanks of raw liquors that such control should be exercised, but also at the char-filters and systematically and generally throughout the house as occasion requires. Alkalinity and acidity can be readily ascertained in a qualitative way by means of blue and red "Litmus Paper"— the blue paper turns red or pinkish when dipped into an acid liquid; the red paper blue or bluish when wetted by an alkaline solution. The degree of color change serves as a rough indication of the intensity of the reaction. For example, if a piece of blue litmus paper—"test paper"—should turn distinctly red, or rather, to a bright salmon pink, it would mean that the liquid just tested is decidedly acid. Reversely, red litmus in changing to blue indicates alkalinity, the degree of this alkalinity may be roughly approximated by the intensity or depth of the blue color. Whenever either blue or red litmus paper is but slightly affected a practically neutral liquid is under observation—it may be either very slightly acid, or alkaline, but may in this case be regarded as neutral if the paper is known to be of standard sensitiveness.

Litmus Paper.—Litmus papers are made by dipping pure, white filter paper into a properly-prepared infusion of a blue pigment, litmus or lacmus obtainable from many species of lichens. The paper is impregnated with the sensitized infusion of litmus, is carefully dried, cut into convenient strips (1½ inches x ¼ inch) and preserved in air-tight boxes.

The test papers should be exposed as little as possible to moist air, as any traces of alkaline or acid vapors in the atmosphere would react upon them and destroy their sensitiveness.

Litmus is only one of the many pigments and chemical indicators that exhibit distinct color changes when acted upon by either acid or alkali. But litmus paper is almost the exclusive indicator used in the sugar industry when the tests to be made are in the hands of skilled labor only. It is of course, rapid, requires little skill in application, and under the eye of an observant workman or foreman, the changes and shades assumed by the paper used become familiar and a fair estimate of the reaction of the liquid may be formed. Litmus paper, then, should be liberally supplied wherever its services may be required and a constant check maintained through its aid. Workmen should be instructed by the chemist in the details of its use at the various stations and informed, as clearly as possible, what interpretations are to be put upon the shades of the color changes. Preferably instruction should be given by testing samples in which the alkalinity and acidity has been definitely determined by chemical analysis. When dark syrups are to be tested certain precautions and manipulations are to be observed, otherwise the indications, or rather the inference drawn from the indications may be misleading to the uninstructed operator.

CHEMICAL TESTS AND LIQUOR REACTIONS

With reference, however, to qualitative reaction determination of liquors and syrups at the treating stations and the char-filters, an accurate system should be adopted which can be carried out satisfactorily by the control chemist.

The scope of this work does not include the discussion of laboratory methods of analysis, inasmuch as such methods are to be found in chemical and technical hand-books, but statements made in regard to the important matter of regulating the reactions of liquors and syrups are so frequently expressed in such vague and indefinite terms as to be more confusing than helpful. Therefore a few words on the subject will not be out of place, especially as all references in this book to "degrees" of acidity or alkalinity are based upon the following:

Method of Recording.—Alkalinity is indicated by the minus sign (—) and acidity by the plus sign (+) in accordance with chemical practice. The "degrees" are expressed in digits, in terms of CaO, and mean in the case of alkalinity the number of milligrams of CaO in excess of neutrality which are present in one liter of the liquor, and in the case of acidity the number of milligrams of CaO which would be required to neutralize one liter of the liquor under examination.

Example.—If 10 cc. of liquor required 0.2 cc. N/28 H_2SO_4 for neutralization, the result would be recorded as —20, because: an imaginary normal solution of lime would contain 28 grams of CaO, or 28,000 milligrams in a liter; a 1/28th normal solution (written N/28) contains 1,000 milligrams in a liter or one milligram per cc.

Therefore 0.2 cc. would contain 0.2 milligram of CaO.

But in the example only 10 cc. were taken for titration and the results are to be expressed "per liter;" therefore $0.2 \times 100 = 20$.

Of course any convenient strength of standard solution may be used for titrating, but N/28 is probably the most convenient because it represents one milligram CaO per cc., is dilute enough to permit of accurate titration, and strong enough to indicate a sharp end point in all but decidedly dark liquors, etc.

It here becomes imperative to speak of indicators. Litmus and lacmoid are true neutral indicators (H x 10^{-7} : OH x 10^{-7}) and are quite suitable for white liquors, but owing to the difficulty of seeing the end point, as indicated by the production of a violet tint, *i. e.,* half-way between blue and red, they are seldom used.

Phenolphthalein is a great favorite, but it must not be overlooked that this indicator is extremely sensitive to acid, and therefore does not indicate the true end point.

The authors have found "Brom-Thymol-Blue" (Dibromthymolsulphonephthalein) a very useful indicator, which is however somewhat supersensitive to alkalies, but far more reliable than phenolphthalein, and producing a far sharper end point.

Experiment has shown that a solution which is neutral to litmus is —20 to Brom-Thymol-Blue and +40 to phenolphthalein.

All "degrees" quoted in this book of course refer to true end points.

LIQUOR REACTIONS

At the treating tanks solutions are usually required to be alkaline, and customarily range from —10 to —40.

At the char-filter "running gutters," acidity and neutrality are required, and in the daily reports may appear as for B liquor +10; A +20; refiltering liquor +50, etc.

In the preceding paragraphs a few of the more distinctive control methods have been indicated, but in a general supervision of the interrelated factors of all operations many analyses, measurements, tabulations and quantitative and qualitative tracings of the materials in movement to their final goal will have to be made.

From time to time special investigations, relative to studies of peculiar and local problems will obtrude themselves, so that a well-equipped research laboratory is always a valuable adjunct to a refinery.

Control of fuel, steam regulation and its economic distribution, etc., are so much modified by local conditions and requirements, that systems involving these problems must be left to the management of this important department. In most establishments the technical and engineering staffs will cooperate in this respect.

Finally, the collection, interpretation and calculation of the significance of control data, gathered during a period of activity, will be illustrated after the next chapter on Char-house Management will have, within the scope of this treatise, completed the sequential discussion of sugar refining.

pH DETERMINATION

As regards a more accurate and basically scientific control of liquor reactions, the pH determination is beginning to find practical application in sugar manufacture and refining, and it may be advisable for the superintending staff to cooperate with the chemist relative to maintaining sugar liquors and syrups at certain pH values.

The extent to which such control may be carried is a matter of specific determination, according to the needs and methods of each establishment, and, therefore, the details of working with pH values is to be decided by the operating staff and the chemist.

There are, however, quite a number of articles to be found of a practical nature in sugar journals (Vide, *International Sugar Journal,* 1923, page 88, and *J. A. C. S.,* Vol. 42, No. 4, p. 742), and many other publications. For a general study of the principle and applicability of the potentiometer and hydrogen determination Dr. W. M. Clark's work, "The Determination of Hydrogen Ions" may be consulted with great advantage.

It is now generally conceeded that operating with pH value of 6.5 to 7.0 a minimum inversion is experienced, but whether this value can be maintained at all stations of a refinery can be determined only by observation of prevailing conditions and the nature of the final products required.

In this respect "Inversion Losses in Cane Sugar Manufacture" (*Industrial and Engineering Chemistry,* Vol. 17, page 51, 1925) deals with many aspects of the refiner's problem of maintaining liquor reaction.

CHAPTER IV

CONTROL

CHARS, QUALITY AND COMPOSITION OF WORKING CHARS

Among the numerous substances that have been practically applied in the sugar industry, there is none that approaches animal black or char as an efficient decolorizing agent primarily, and, in a secondary direction, as an absorbant of organic and inorganic bodies associated with sucrose in raw sugar. Not only in its general purifying effect is char unique among its congeners, but also in its granular form it can be more readily handled, revivified by heat, or otherwise—in a supplementary manner—have its pristine qualities in a large measure restored for repeated cycles of usefulness. In short, then, through the fact of its preeminent position in refinery operations, a fairly extensive treatment of char problems in general together with illustrations of a practical nature are essential here.

Historical Sketch of Char and Amorphous Blacks.—A short historical sketch of amorphous blacks—carbon—regarded as decolorizing agents will naturally contribute to a study of the peculiar power exhibited by carbon when in that physical condition. Furthermore, it has recently been shown that in extremely fine state of division *and dispersed upon some inert material,* effectiveness of amorphous carbon is enhanced, it should be remembered, however, that the amorphous black alone is the active agent in a substance so produced.

The employment on an industrial scale of substances containing a large percentage of amorphous carbon as a decolorizing agent, is an art that has been developed during the nineteenth century. Lowitz is credited with having first recognized, in 1785, the decolorizing power of carbonized plant tissues, and a few years later wood charcoal was suggested as a decolorant for sugar solutions. But residual carbonaceous products obtained from the destructive distillation of animal tissues presently superceded those of plant origin, as the power of the former soon gave evidence of their much greater efficiency as decolorants. This superiority was determined in 1811 by Figuier.

The eventual introduction of animal black in a granular form, and therefore more readily revivifiable, secured to this material the most important place among the decolorant blacks of commerce.

In passing, it may be remarked that the convenient word "char," will be used in the place of bone-black, animal black and the like.

It is a curious coincidence that the early studies on the decolorizing and purifying properties of char, and the amorphous blacks in general, were almost coeval with the development of the beet-sugar industry of western Europe during the first half of the last century, and the first investigations on char utilization were nearly all more or less intimately connected with the then rapidly progressing industry of beet-sugar manufacture and refining.

Initially the different decolorizing blacks were employed in a powdered form, and in as fine a state of comminution as practice permitted, even charred bones (char) were then ground to a fine powder and added to the sugar solutions, the liquid heated, agitated and the char strained off and discarded. It was not until 1825, however, that the possibility of regenerating the decolorizing power of a several times used chardust was shown to be practicable, but, of course, handling of such an immobile, dusty substance presented many difficulties. In 1828, however, Dumont suggested the granular form of char, and thus a true filtration was made possible and the handling and the revivification greatly facilitated. Again, another form of bone carbon was tried. The charred bones were ground, digested in hydrochloric acid; the soluble salts washed out with water and the residual carbonaceous matter was known as "Purified Animal Black." This so-prepared black cost something like two shillings per pound in England at the time.

Generally speaking, nearly all blacks of a non-graphitic nature, possess some decolorizing power, and the phenomenon is so marked in the best of them that it is no matter of wonder that a large number of investigators should have entered this fruitful field of research, and through their labors the industrial application of char expanded very rapidly. Not alone in the sugar industry did char find profitable employment as a decolorant, but also upon solutions of glucose, liquid paraffins, molten or liquid fats and on crude glycerol amorphous blacks and char were proved to act as powerful color adsorbants. Incidentally other substances are retained that may be associated with the principle constituents of a liquid or in the solution of a raw material. Furthermore, it was presently observed that char does not only adsorb color from complex solutions, but that it will retain—and frequently with great avidity—certain mineral and organic salts and a large number of organic compounds of diverse chemical composition.

The occlusion of gases by blacks is another phenomenon of importance, as under certain conditions of adsorbed oxygen, char and other blacks, will *darken* colored solutions instead of bleaching them.

It may be said, then, that a large amount of empirical information had been gained by the comparatively rapid entry of char into a number of industries, and that at the end of the first third of the last century the main problems of a practical nature had been fairly worked out.

As has been pointed out, digestion preceded filtration as an industrial practice. Revivification methods for granular char were, however, for a long time cumbersome and frequently highly offensive. Usually before heat regeneration, the char was subjected to washing with caustic soda, followed by a wash with dilute hydrochloric acid to neutralize the last traces of residual soda. Not infrequently, imperfectly washed char was taken from the filters, spread out in layers of about 2 feet deep and allowed to ferment. Along with fermentation went putrefaction of the nitrogenous compounds residual in the used char, so that an exceedingly evil-smelling material has to be handled. The fermented char was later washed with acid and flowing water, and after being sufficiently dried, passed through kilns for revivification. These treatments of char are now very seldom practiced, except in cases where, through negligence some constituent, removable by these methods may prove useful. Thorough washing with hot water followed by drying and heat revivification are usually effective in maintaining the life of a char to the limit of its usefulness in refinery practice.

In the direction of what may be called the "selective adsorption" of char relative to organic and inorganic salts, Walkoff, of Magdeburg, about 1870, determined adsorption figures obtained by investigation on an industrial scale. Walkhoff's observations have, in the main, been corroberated by other investigators upon this important point.

This sketchy review of char utilization brings us, then, to within fifty years of our time and it may be said that so far as sugar refining is concerned, comparatively little progress has been made, both in applied and pure chemistry of amorphous blacks. Various incidental processes have been suggested with the view of conserving or enhancing the quality of "working chars," *viz.*, those chars in the cycles of refinery operations, but these suggestions have contributed only in a small way to the advancement of char chemistry. Indeed, in a number of directions there are to-day many questions in char chemistry that need investigation as much as in the days when this material was being adapted to the various industrial interests. Conceding the immediately practical data to have been fairly dealt with there is nevertheless a vital question for which no satisfactory answer has yet been found. The remarkable decolorizing power of amorphous black having been established, and the condition of maximum efficiency fairly known, it is quite natural to expect that interest should presently become centered upon the question as to the cause of this peculiar property of blacks. It is then a matter of surprise that after nearly a hundred years of practical acquaintance with decolorizing blacks, the scientific world should not yet be in accord upon this point. "Why does amorphous carbon decolorize?" The interrogation mark, after a half century of work, has not become any smaller. With the exception of a few more or less disjointed efforts the problem still stands calling for original and thorough research.

Work, of course, has been done with the aim of answering this question, but as yet it cannot be said that any convincing answer has been given, nor do the researches made cover all the problems in the case.

This chapter being largely limited to practical questions, the chemistry of chars will be touched upon only in so far as it is essential to working requirements. There is hope, however, that the recent changes in the concepts of the nature of solution of salts and the recognition of the colloidal condition of many coloring matters when in solution, may presently yield a rational basis for a chemistry of decolorizing blacks.

Another contributing and cognate factor of this phase of char problems may presently develop through the recently created interest in the manufacture of decolorant blacks other than char.

With the incoming years of the present century, say within the last twenty years, a large number of blacks have been made, all more directly resulting from the endeavor of finding a cheap substitute for char. Most of these blacks are used in a sooty or extremely fine-powdered form, and for the regeneration of their power chemical reagents, such as soda and hydrochloric acid are used, thus reverting somewhat to the old method applied to char proper.

Lastly, during the World War, blacks were sought that might be useful as gas adsorbants, and thus promising researches on the "activation" of blacks were instituted. Summing all these facts, there is hope that the pure chemistry of decolorant blacks may be advanced within the next few years.

In Part II, Chapter II, on Purification, the system of char-filters and the utilization of char relative to liquor and syrups to be filtered was treated in an elementary manner and dealt with in so far only as was necessary to the linking of this department with the preceding Preliminary process, and the succeeding Production, Chapters I and III, respectively of Part II.

In the description of filtration in that chapter, the liquors were considered as entering filters that had been recently filled with appropriate chars for the service required of them. After the cycle of filtration had been described, from settling to sweetening-off, of the filters used, the liquors were followed to the vacuum pan, etc. The filters themselves were left filled with exhausted char that must be washed, blown, drawn and sent to the kilns. Obviously, then, there remains to be dealt with those operations involving the handling and treatment of "working chars" from the time that the exhausted char is being washed to the time when it has been revivified and is again ready to reenter an empty filter for a succeeding cycle of usefulness. In this sense, then, this char manipulation is purely an adjunctive operation, while the char-house management, as will presently appear, demands a large percentage of the control work of the technical staff.

OPERATION OF "WORKING CHAR" SYSTEM

In the char-house there are two systems so intimately connected that if either one or the other should be interrupted and the necessary regularity of operation in any way become intermittent, both will be thrown out of full efficiency proportional to the nature of the departure from the normal condition required of the system.

One of these systems has for its immediate object the char distribution and supply; the other, in an analogous manner, deals with the liquors and syrups to be filtered. Both systems are synchronized in all their phases so that the total volume of sugar solutions shall be filtered through a definite, predetermined quantity of working chars within a certain period of time, say, every 24 hours. In fact there is in this department not only the necessity of synchronizing char and liquor systems, but these must also, as it were, be intersynchronized for complete and efficient service.

THE CHAR SYSTEM

Beginning, then, with the chars and their distribution, it is usual in present practice to have three qualities of char charged with the filtration of raw liquors and syrups according to their purity and color content. Besides these three chars there should always be on hand a quantity of new char, ready to make up the material losses and to maintain the standard of the working chars. It is usual to fix, from time to time, the quantity of new char to be added to the working chars.

As it is essential in the char-house control that the purity of the liquors and syrup be known, so also must the quality of the char be established, and periodic analyses and tests made with this end in view. Some tests should be made every 12 or 24 hours, others every 2 or 3 weeks, while a complete analysis should be made of the working chars every 2 or 3 months. Experimental filtration tests and the determination of other interesting data will belong to char chemistry and laboratory control relative to this material, and only under exceptional cases will such enter into the question of control and management of the char-house.

To have on record data establishing the quality standard of working chars, four main examinations are to be made; these are:

I. Chemical Analysis.
II. Grain Analysis.
III. Specific Gravity in Bulk (weight per cubic foot).
IV. Daily Tests of Revivification.

COMPOSITION OF CHARS

I. **Chemical Analysis.**—As previously intimated the chemical composition, *per se,* of a char gives only comparative aid in determining its filtering value. There are two or three constituents of the char, however, that should not exceed certain well recognized amounts.

Take as typical, the analysis of a new char should approximate to:

Carbonaceous matter	8	to	12	per cent.
Sand and insoluble matter	0.5	to	0.7	per cent.
Iron, as ferric oxide	0.2	to	0.3	per cent.
Calcium carbonate	6	to	8	per cent.
Calcium sulphate	0.05	to	0.20	per cent.
Calcium sulphide	0.01	to	0.05	per cent.
Calcium phosphate	about 80			per cent.

Water may range from 2 to 6 per cent, according to condition of storage, etc.; from a humid atmosphere char may absorb up to 7 per cent or even 8 per cent of water.

Setting against the above analysis, three made upon chars that had been in use some time we find.

	No. 1	No. 2	No. 3
Carbon	10.90	12.00	11.70
Sand, etc.	0.80	0.80	0.80
Iron as ferric oxide	0.54	0.56	0.56
Calcium carbonate	4.13	3.92	4.31
Calcium sulphate	1.06	1.13	1.18
Calcium sulphide	0.12	0.14	0.12

Char No. 1 represents the least used; No. 3 the most used, while No. 2 is of intermediate quality. Comparing the analytical data, it will be noted that the ferric oxide has doubled and both the calcium sulphates and sulphides have increased tenfold. Calcium carbonate has been reduced by 50 per cent.

But it is upon the three first-mentioned constituents that attention should be centered, as any sudden increase shown by any of them would be an indication that the char in question is not receiving proper attention and therefore subject to unwarranted deterioration. Nor should calcium carbonate fall below, say 3 per cent in the older chars. Inasmuch as the presence of calcium carbonate is desirable the sulphates and sulphides are distinctly undesirable.

Calcium sulphate seems to be a natural constituent of the raw bones and is thus always present in small amounts in new char, but the sulphides are derived from the sulphates during charring of the bones and also during revivification of the char in the kilns. The sulphides are produced by the reducing action of the carbon in the char, thus:

$$CaSO_4 + 2\,C = CaS + 2\,CO_2.$$

The progressive accumulation of the sulphates, and in consequence of the sulphides also, is due to the fact that char adsorbs calcium sulphate very readily and retains it tenaceously. This is one of the chief reasons against sending syrups, that have been sulphured, for refiltration through any working chars.

Calcium carbonate disappears since it is readily soluble in all acids, and sweet-water running to acid reaction dissolves this salt which is carried away in it and in the waste-waters.

Iron in new char is chiefly due to nails, etc., mixed with the raw bones and accumulates in the used char by adsorption from sugar liquors, and abrasion from machinery.

Carbon, or more properly speaking, "carbonaceous matter," tends to accumulate, and if no efforts be made to remove it, the percentage of this element may rise to 16 -18 per cent in the older working chars. If then periodic determinations of carbon indicates that accumulation approaches a limit that may be set by the management, some of the well-known "decarbonizing" methods may be resorted to. This question will be taken up under kiln work.

On the whole marked changes of composition of working chars—beyond that incidental to natural "ageing"—in very infrequently encountered.

II. **Grain Analysis.**—Under grinding and screening of burned bones, it was suggested that a refinery making its own char should range its screens to meshes 10 to 50. But when char is to be bought 16 to 30-mesh should be specified.

In discussing grain and grain analysis of chars it will presently become apparent why this distinction should be made. As stated in the historical sketch, it was at once recognized that the finer the state of division the higher the decolorizing power of the char, and the limit of comminution was fixed by the fact that the finer the char the more difficult filtration became; also recovery of sugar from the used char of finer grain is not so readily brought about. Eventually, then, the now accepted size of grain —16 to 30-mesh—has become the general standard upon which refiners buy their black.

Very few beet-sugar refineries now use char, but in that industry a rather coarse-grained char was for a long time in vogue —grains the size of small peas being usual. Since comparatively little color had to be removed from the raw-beet sugar, the refiners naturally use this large grain with advantage—losing less by attrition and sweetening-off with a minimum of water.

If however, the refinery produces its own char there is no reason why advantage should not be taken of the 10 to 15 per cent fine grain unavoidably formed during the grinding. This fine grain is very active, and when used as presently indicated, represents so much extra yield from the raw bones as char producers. And when grain analyses are made frequently enough there need be no fear that too much of this finer grain will be allowed to accumulate; in any event it can readily and speedily be removed while the char is performing its functions in the char-house.

Grain analysis should be made upon the new char as it comes from the mill so that any break in the screen may be detected or any block be rectified, and of course, to assure a uniform product. Working chars are examined for a like reason, since the accumulation of too much fine grain, or dust also may render the filtering and other operations troublesome.

The screening for this analysis is done through a set of standard screens, five in number, *i. e.*, 16, 20, 30, 40 and 50-mesh. The screens are "nested" one above the other and 200 grams of char is placed on the upper—16-mesh—screen. Under a uniformly observed shaking the grains of different sizes remain on their respective screens and are eventually placed in a balance and weighed, and the weights calculated to percentages. Mechanical shakers are, of course, more uniform and reliable.

As examples of 16 to 60 and 16 to 30-mesh new chars, the subjoined figures may be considered as typical.

Grain		16–30	16–60
Remaining on	16	74.00	35.00
Remaining on	20	17.50	24.00
Remaining on	30	6.20	30.00
Remaining on	40	0.40	8.00
Remaining on	50	0.40	1.00
Through	50	1.50	2.00

Summing the percentages of grain on 16, 20 and 30-mesh screens, we get as remaining on 30, 97.7 per cent of the 16 to 30-mesh char and 89 per cent of 16 to 60-mesh. An advantage is to be noted in this, that in the 16 to 60-mesh product, 11 per cent fine grain will for awhile be serviceable as against 2.3 per cent in the 16 to 30-mesh char.

A new char with rather an excess of fine grain gave upon analysis:

Grain		16 to 60
Remaining on	16	8.52
Remaining on	20	10.32
Remaining on	30	27.00
Remaining on	40	27.48
Remaining on	50	14.72
Through	50	11.90

Working chars gave:

		Char.		
Grain		No. 1	No. 2	No. 3
Remaining on	16	11.4	6.8	8.1
Remaining on	20	19.8	16.4	16.9
Remaining on	30	29.9	34.1	27.2
Remaining on	40	17.3	21.4	27.7
Remaining on	50	11.9	12.7	13.8
Through	50	8.5	8.0	4.7

III. Specific Gravity in Bulk. (C. F. W.)—The weight of a cubic foot (C. F. W.) of char gives a ready though rough indication of the age or relative quality of char, particularly when the weights of char of similar comminution are compared. Indeed, very little information can be drawn from the c. f. w. of chars of distinctly different grain structure.

The increase of the c. f. w. figure is due to the fact that chars cannot be washed entirely free of substances they have adsorbed from the sugar solutions. Upon revivification, the, what might be termed, permanently adsorbed matter remains lodged upon the walls of the bone tissue, but without increasing the bulk of the char and therefore the c. f. w. figure is due to the accumulation of insoluble, or slightly soluble lime salts and also to the rising percentage of carbon. The excess carbon is derived from the permanently adsorbed organic matter, some of it, no doubt, in combination with mineral bases. Under the heat of the kilns this organic matter is decomposed with the production of residual carbon, quite analogous to that produced in char production from bones. But the original carbon deposited upon the tricalcium phosphate of the bone has high decolorizing power, and any further addition of carbon, inert and graphitic, may be looked upon as excessive, which, indeed, it is. This secondary deposit of carbon, therefore, must thicken the carbon wall on the tissues, diminish the diameter of the capillary tubes and gradually close up the reticular spaces of the bone tissue. This, of course, means that the char, bulk for bulk, is losing surface and gaining weight. But the enormous surface of char is due to the reticular nature of the bone tissue which is certainly one very important factor in char as a decolorant. Loss of surface is therefore just that much loss of function.

The c. f. w. of a char may then be considered a fair indicator of permanent and relative exhaustion of a char.

Relative to the c. f. w. of the char reviewed under (II) Grain Analysis:

16-30 new char would be	40 pounds per c. f. w.
16-50 new char would be	42 pounds per c. f. w.
No. 1 working char would be	52 pounds per c. f. w.
No. 2 working char would be	58 pounds per c. f w.
No. 3 working char would be	60 pounds per c. f. w.

The weights of Nos. 1, 2, and 3 represent maximum c. f. w.

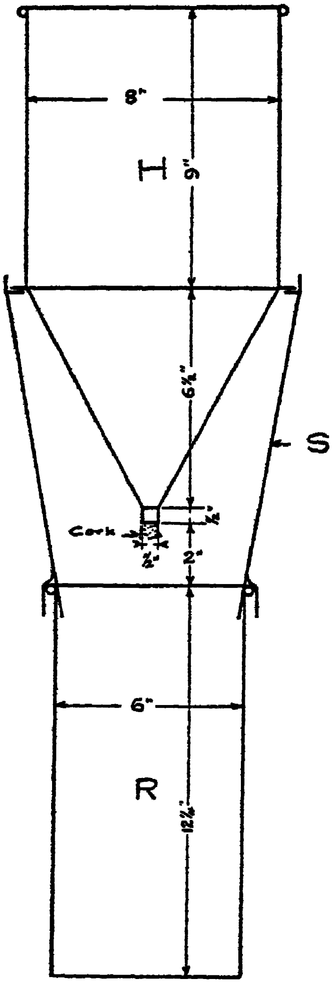
In some cases when a char is to be used as a preliminary filtrant of black strap or low-purity green, raw or refined, a char may be employed having a c. f. w. of from 70 to 72 pounds.

The c. f. w. of a char may be roughly determined by weighing a cubic foot of the char filled into an appropriate cubic foot measure, under a determined number of taps produced by uniform tilting of the vessel. Another and accurate procedure is to pour the char into a measure from a determined and constantly observed height.

A simple contrivance such as shown in the accompanying sketch, Fig. VIII, has been found to give very consistent results, agreeing closely with the weight of chars as run into filters from the hoppers, which weights were actually determined experimentally.

H is the hopper in which the char to be weighed is filled. It is provided with a funnel-shaped bottom, F, and a one-half inch circular outlet which is corked to retain the char.

S is an iron support resting on the edges of the receiver R, in such a manner that the end of the outlet at F is 2 inches above the



Apparatus for the determination of C. F. W. of Chars.
Fig. VIII.

level of R. The dimensions given for the apparatus are convenient as they provide a factor of 0.01.

A c. f. w. determination is made in the following manner:

H having been corked is completely filled with the char to be tested, and placed upon S.

R is counterbalanced and H and S are set in position upon it. The cork is then withdrawn and the char runs into R until the cone formed by the running char reaches the outlet and automatically stops the flow.

R and S are then gently removed, and the char in R is leveled with a straight edge, all tapping or shaking being carefully avoided. After leveling, the sides of R may be tapped, causing the char to sink and making it more convenient to handle the vessel.

The weight of the char in R is then taken in grams, and the figure obtained multiplied by 0.01 the product being the weight per cubic foot of the char.

Example:

	Weight in grams found	C. F. W.
A char	5,043	50.43
B char	5,591	55.91
C char	5,916	59.16

IV. Daily Tests to Check Revivification.—This test is made upon a daily average sample of the revivified working chars. Small quantities of char are taken every hour or so from the conveyor belts, or any convenient place, and put into a covered vessel. At specified times the average sample is taken to the laboratory to be tested.

The sample is carefully mixed, about two ounces are placed into an absolutely clean casserole and flooded with 50 to 75 cc. of a pure caustic-soda solution—15° Brix is usual. The contents of the casserole are then brought to a boil over a Bunsen burner, and after a few minutes' boiling the liquid is poured off into a clear, white, clean test-tube and set aside for a few minutes. Presently the finer grains of char, that have run into the tube along with the soda solution, will have fallen to the bottom and the test liquor may now be inspected.

The object of the test is to determine whether or no the char has been properly revivified, *i. e.*, whether all the organic matter the char had adsorbed during filtration has been destroyed. If this condition has been attained the test liquid in the tube will have a very slight yellow tinge, but any shade approaching a straw tint should be regarded as an indication of insufficient revivification, and that there is present in the char tested a further removable quantity of organic matter. A test liquid of a yellowish brown tint is indicative of poor work, and the char has certainly, in this case, not been properly burned and poor filtration will be observed in the filter in which the char in question may be used.

On the other hand, a distinctly white test liquid is not to be considered as an exhibition of good revivification. In such a case the char may have been overburned, which is of no advantage in filtering and involves the destruction of the char itself, tending to the decomposition of a certain nitrogenous carbon combination associated with the carbon in the char. This "nitro-body," along with the carbon is a factor of efficiency in the black.

The rational of the test is simply this: The strong caustic solution attacks any organic matter present with the formation of a yellow color and therefore the depth of the color observed will be an indication of the presence of such organic matter. The carrying out of this test is, of course, exceedingly simple, but there is one essential precaution to be observed, *viz.*, the sample of the char must not be touched with anything that might convey "dirt" or any organic matter to the sample.

CHAPTER V

CONTROL OF CHAR SYSTEMS

With the aid of the illustrative Diagram X, the migrations of the working chars may be followed and the successive operations of a cycle of char work coordinated with the liquor and syrup systems from the time these solutions enter the filters and leave the char-house by way of the running gutters.

If three qualities of working chars are to be operated, then three such systems will have to be installed. They should be designed for independent operation, since, for instance, a battery of filters served by one grade of char may have a longer or shorter cycle of filtration than another.

As the three-quality char system is now almost exclusively operated, such a system only will come under consideration. For special purposes a fourth-quality char is sometimes installed, but this is not usually a part of the regular operations of a filter-house.

For brief reference the same abbreviations will here be used as in Part II, Chapter II. In matter of quality, A char is to be the best; B of intermediate quality and C of the poorest, and each will, of course, serve its respective set of filters as indicated in Diagrams V and XI, and textual matter relative to filtration.

During the running of a filter house there is in the first place to be provided ample char of each quality to fill the system. A receiver, R; the filters F; the dryers, Dr and the retorts *r* and the coolers *c* of the kilns K, must all be filled, or be delivering their full quota of char as operations require.

The number of each grade of char-filters having been decided upon, and the length of their cycle determined, the rate of char movement is fixed in a manner presently to be explained, one filter in the battery should always be ready to receive the revivified char that has accumulated in R, between the time of a previous packing of a filter and the next one.

Since a cycle of filtration may be followed by referring to Chapter II, Part II, the char movement only will now be considered.

Char House System.

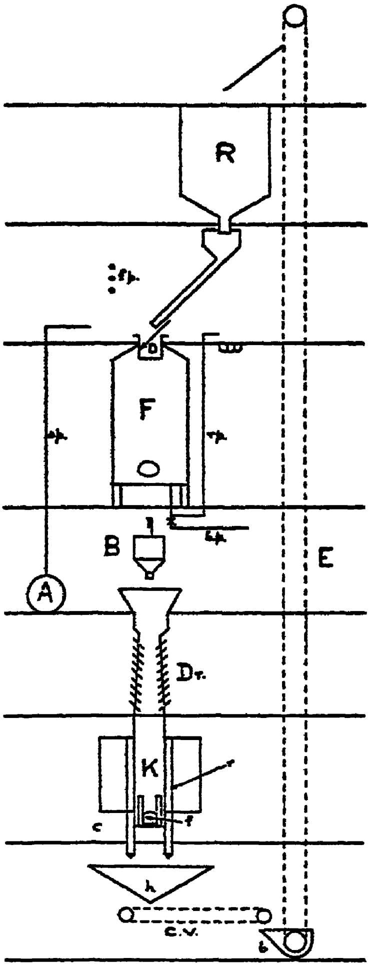


Diagram XI.

Beginning, then, with an empty filter, F, and a full receiver, R, which has the capacity of a filter—about 1,000 cubic feet—the char can be run into the filter from R in from 60 to 70 minutes. There it remains during the filtering cycle, and is during that time in the char system only in so far as it will be necessary to see that the filter be ready at the calculated time to be "drawn" when this char will be sent on its way for revivification.

Char Pumping.—After the char in this filter has done its work; has been sweetened-off and pumped, the man-hole *m* is opened and the wet char drawn out. The last step of char treatment in the filter, *viz.*, pumping, has for its object the forcing out of the adhering wash-water and also to reduce the temperature of the char so that it can be handled. With this in view, the head of the filter is connected with the air pump, A, by way of the pipe system *ap* and a current of cold air pumped through the char. The moist air—and at first some hot water—finds exit at the blow-off pipe *bp* at the bottom of the filter.

Pumping should be maintained with a minimum pressure of 15 pounds—20 pounds is better—for at least 3 hours, or longer f time and capacity of the house permits. Dense chars, such as the C char, will require 5 hours or more of pumping. But even after complete pumping, the char drawn will contain 17 to 18 per cent of water, and will not, therefore, flow out of the filter man-hole. It has to be drawn out by hoeing, and conveyed to the drier.

The manner of this conveyance is largely a matter of local necessity, but it may be added that belt conveyors are generally admitted to be troublesome. The wet char adheres to the belt, s carried about with it, and tends to be ground and otherwise destroyed. Bucket conveyors, buggies on properly placed overhead rails, etc., are the most convenient methods for handling wet char.

Char Drying.—From the filter the wet char finally finds its way to a hopper above the drier floor and slowly descends into the drier, *Dr*, and is heated by the hot flue gases from the kiln fires. Issuing from the drier it is possible that the char may not contain more than 10 per cent water, but if the ventilation of the drier floor is not ample, the moist char about to enter the upper end of the retorts *r* may still hold 14 to 15 per cent of water. Too much stress cannot be therefore laid upon the necessity of providing a lively circulation of dry air, especially when the type of drier used is dependent somewhat upon surface evaporation into the drier flat. Direct experiment has shown that a current of air driven against the descending char on the face of the drier, may reduce the water content of the char by 2 to 3 per cent; a very material consideration when it amounts to sending 20,000 to 30,000 pounds less water onto the top of the retorts.

As regards the construction of driers, the scope of this work, only permits the general statement that they all aim at distributing the char in thin layers and with easy flow over as large a heating surface as possible. The flue gases pass behind plates upon which the char moves, and some driers have the very desirable system of fan-driving the air through the driers or over the surface of the char on the driers.

Revivification.—This important feature in char control is carried out in a kiln which may be described as a rectangular, fire-brick lined room provided with arches and flues for the distribution of the heat from the fire place *f* in K.

The retorts of cast-iron are elongated O-shaped tubes in cross-section, 3 inches x 12 inches and between 8 and 9 feet long. They are ranged vertically in double rows on each side of the grate, so that the hot gases pass around them and upward, passing by way of the drier to the chimney. A double kiln holds seventy-two retorts, so that 140 to 150 cubic feet of char is slowly descending through them in the process of revivification.

With char entering the retorts with not more than 10 to 12 per cent water, each retort may attain a maximum capacity of 22 cubic feet of revivified char daily. If, however, the moisture of the char is high, the kiln capacity falls, and frequently not more than 15 to 17 cubic feet will be the 24-hour yield per retort. Nor should superdriving of kilns ever be indulged in; excessive firing not only tends to local overheating of the char in the retorts, but the life also of the retorts is considerably reduced and damage will result to the kiln arches and flues and necessitate more frequent rebuilding of these. Taking all conditions into account, uniform firing, means uniform heating of the char, together with regular supply of wet char and the withdrawal of the revivified will in the end bring best average results in a period of refining.

From the foregoing, by implication, one would infer that a fixed temperature of revivification is to be desired. Both as to economy of fuel and the preservation of the char no higher degree of heat should be applied than that necessary to destroy the adsorbed organic matter. Seven hundred to 800° F., of the char itself, has been established as ample for this purpose. But kiln design, unfortunately, does not readily lend itself to placing of pyrometers where they would give definite information of the actual temperature of the char passing through the retorts. Lacking this aid to heat control, the superintendent staff is usually charged with periodic inspection of the retorts. These may be viewed through peep-holes. Experience and judgment must be relied upon to keep conditions right in this respect, and it is then a matter of insisting that the kiln firemen maintain a fire that will keep the retorts at a dull red heat—cherry-red; any conditions approaching a bright red will bring about evils pointed out in the preceeding paragraph. Some establishments have, however, installed pyrometers, usually of the recording type, but even in such cases the proper pyrometer temperature has to be determined by experiment, as the instruments cannot be placed to record the heat of the char itself, but only the temperature of some neighboring flue. From the pyrometer data and the revivification tests, the degree to be maintained is determined, so that at best only indirect control is to be expected from such an installation. Usually a temperature of 900° to 1,000° F. of the gases *entering* the driers will be found sufficient.

Coolers.—The external continuation of the retorts are black, sheet-iron tubes, known as coolers, and are of the same cross-section as the retorts. They are 8 to 10 feet in length.

The red-hot char passing down from the retorts descends through the coolers *c* and by the time it has reached the lower end has sufficiently cooled to be admitted to the air, and passing through the hopper, *h,* falls upon the conveyor *cy* is carried to the elevator E to be taken to R, completing the cycle. The entire flow of the char through *Dr, r* and *c* is regulated by a mechanical device attached to the bottom of the coolers and operated by a driving machine, technically known as a "clock." The clock can be adjusted to move the drawing mechanism at a rate that will deliver the char in desired quantity demanded by the system.

Loss, Attrition, Degeneration and Distribution of Chars.—Working chars constantly being moved and used throughout the system as just described, naturally fall from their original qualities in two ways. By repeated adsorption of organic matter and salts unextractable from the char, degeneration goes on continuously. Again, by attrition a certain amount of char-dust is formed; small particles are constantly being broken from the larger grain and the smaller reduced to a size necessitating their removal from the body of the char. Losses due to leaky retorts, driers and spillings from belts are small and quite unusual.

But attrition and degeneration must be made good by a regular introduction of new char into the system. The quantity of new char to be added daily to the working chars will depend largely upon the nature of the raw sugar and the percentage of softs to be made. Soft-sugar production implies the filtration of a larger volume of raw syrups, therefore a greater strain is put upon the char. Attrition as well as exhaustion of the chars will be greater since the filter cycles will be shorter and revivification more frequent relative to a unit amount of raw sugar refined. If, say 30 per cent softs are to be made, it would be necessary to introduce about 1 per cent of new char upon the amount of raw sugar refined daily; on the other hand, should soft production fall to 10 per cent, not more than one-third of 1 per cent of new char would be necessary.

Whatever quantity of new char may be introduced, however, it is naturally added to the A quality which obviously should be the recipient of the fresh material. But the relative bulks of A, B and C chars must be maintained so that if *n* pounds of new char be added to A, *n* pounds of A will be sent to B and *n* pounds of B to C.

From time to time if the bulk of C increases beyond the limits set, some of it may be taken out as "Discard Char."

But through attrition fine grain is constantly being formed and must be removed. It will be remembered that a certain amount of fine grain—that between 40 and 60-mesh—was allowed to enter the new char with the standard grain of 16-30, and as the new char is added to A char, there is introduced into this grade a quantity of fine grain—too fine for the filtration of the viscous syrups that are to be passed through C char. But A char, receiving for filtration liquors of high purity only or the refiltering liquors from the B filters, may be used in a finer state of division. B and C chars, however, must be free from this fine grain, which, indeed, has done ample work by the time it reaches the intermediate and lower grades of char; *viz.*, B and C. This fine grain is extracted from the revivified B and C char by diverting a part of each from the conveyors on to and through a Newago screen, 50-mesh. This screening separates the finer grain of the B and C char and keeps their grain structure uniform.

The Newago screens may be supplemented by placing a spilling device in the path of the elevator E and by creating a small cataract of char; a passing current of air will carry off some of the dust which can be deposited in a dust bin.

Magnetic Iron.—Metallic iron also can be removed by placing a suitable electro-magnet in the path of the char on the conveyor. Eighty per cent of the iron in new and A char can thus be removed. Electro-magnet systems are to be found upon the market.

QUANTITATIVE ADJUSTMENT OF CHAR AND LIQUOR SYSTEMS

In the preceeding pages of this chapter the general management of the char system has been under review, and the movements of the char followed through a complete cycle. In Part II, Chapter II, the liquors and syrups were considered in a similar manner, but in neither of these treatments of the respective subjects, has any definite quantitative phase of the systems been considered. In char-house control there is such a large mass of material to be provided for and regulated in its diurnal migration, that a strict quantitative basis must be laid out so that no time shall be lost through lack of material at any stage of the process. Continuous and uninterrupted flow of both char and liquors are essential.

Of course it is to be assumed that any well-designed char-house will have its char systems properly balanced so that conveyors, driers and kilns will be able to supply the required amounts of char to keep the filters operating at their maximum capacity if required. Again, the cycle of A, B and C filters will be determined by the quality and quantity of liquor to be filtered, and it is from the fixing of this cycle that preknowledge of the filtering effect may be obtained. Referring again to the chapter on Purification, last paragraph, it will be noted that a filter cycle falls into seven stages, seven operations. Five of these stages do not vary much as to the time for carrying them out. Operations 1, 2, 5, 6 and 7 may be regarded as adjunctive to the cycle, while 3 and 4 only are direct and hardly ever exceed 65 per cent of the cycle hours of a single filter. When exceptionally pure liquors are filtered over an active char, the cycle may be allowed to go as high as 150 hours and the effective cycle-hours may then rise to 80 or 90 per cent.

The determination, then, of these cycle-hours is of great importance, as upon this the direct work to be expected from the filter is gauged. The time allowed for stages 3 and 4 of the cycle may be varied according to the purity and color of the liquors, upon the rate of filtration, etc. But the remaining stages cannot be deviated from in cycle time to any material extent without eventually seriously impairing the quality of the chars, and consequently, their filtering efficiency, which will presently be reflected in inferior liquors and therefore poor refined products. In short, these operations must not be shortened nor hurried.

If then, we have a battery of filters devoted to a particular char—A, B or C—the filter cycle can be determined by the following simple calculation.

$$\frac{\text{The number of filters in the battery} \times 24}{\text{The number of filters daily to begin cycle}} = \text{Filter cycle in hours.}$$

Example: Say there are thirty filters in the battery and ten are required daily, then

$$\frac{30 \times 24}{10} = 72 \text{ cycle-hours.}$$

But if it were attempted to get 12 daily, other conditions not interfering, the cycle would work out:

$$\frac{30 \times 24}{12} = 60 \text{ cycle-hours.}$$

This cycle would be too short for any liquors and suitable only for very low-purity syrups that would exhaust the char rapidly. Of the 60 hours, about 40 would be required for the adjunctive operations so that only 20 hours would be available for actual running of the liquors. Relative to this, 40 hours should generally be allowed as the minimum time necessary to perform the adjunctive operations, therefore:

$$\text{Cycle-Hours} - 40 = \text{Effective Cycle-Hours.}$$

For matter of convenience, the arithmetical rule may be given algebraic form.

Let

B = Number of filters in battery.
C = Cycle-hours.
N = Number of filters required daily.

Then

$$B = \frac{CN}{24}; \quad C = \frac{24B}{N}; \quad N = \frac{24B}{C}.$$

For example, requiring 70 cycle-hours and fourteen filters daily; what must be the number of filters in the battery?

$$B = \frac{CN}{24} \text{ or } B = \frac{70 \times 14}{24} = 40^5/_6.$$

Say forty-one or forty-two filters.

Forty-two filters would give 72 cycle-hours thus:

$$\frac{42 \times 24}{14} = 72.$$

It would then be decided that the system shall contain forty-two filters in the battery, using fourteen daily and running on a cycle of 70 to 72 hours.

Supplementing these calculations, it is a simple matter to ascertain the average rate of flow of liquors and syrups through the filters, etc. As 43 pounds of solids are contained in each cubic foot of liquor at 55° Brix, the total pounds solids entering the house as raw, divided by this number will give the number of cubic feet of raw liquor and raw syrup to be filtered. To this should be added 20 per cent chargeable to possible refiltration of remelts, etc. If further detailed estimates should be required, *i. e.*, the relative volumes of raw liquor to raw syrup (greens) etc., formulae may be evolved by adapting some of the formulae used for similar purposes in different parts of this work (See Index).

In agreement with the dimensions of the filters postulated in these pages, each filter will hold 1,000 cubic feet of char, and 600 cubic feet liquor will find accommodation in the interstices of A and B chars and 500 cubic feet of the C char.

The hoppers, driers, retorts and coolers of a seventy-two retort kiln will hold from 1,200 to 1,500 cubic feet of char.

If, then, the filter cycle of A, B and C sections of the char-filters has been decided upon, and the number of filters in each section fixed, the clock is regulated to draw the necessary char from the kilns of each section.

If, say, twelve filters per day are to be supplied with char from ten operating kilns, then each cooler must deliver 16⅔ cubic feet of char daily.

Upon these data, then, char-house operations must be begun. The quantitative movements of the char has been established and it remains but to study the liquor as it flows through the filters, delivering filtrates each according to the original purity of material sent upon the particular battery.

The general method of distributing the raw liquors and syrups has been dealt with in Part II, Chapter II, but some further detailed information will be obtained by studying the filtration curves in Diagram XII.

Here there are plotted the purities and densities of two A filters upon which the usual refiltering liquor and C liquor had been run.

The purity and density curves do not begin in this diagram until one hour before water had been put upon the filter. It is to be understood, however, that these filters, previous to this time had been running liquor at the rate of 100 cubic feet per hour and delivering liquor at a steady density and practically little decline of purity. The curve of the diagram starts at the time that a fall in purity showed itself and the exhaustion of the char is in progress. Note how the purity falls although the density remains constant up to the thirteenth hour. The rate of flow was changed as soon as water had been put on at the first hour. The rate of flow is now 50 to 60 cubic feet, as indicated by the

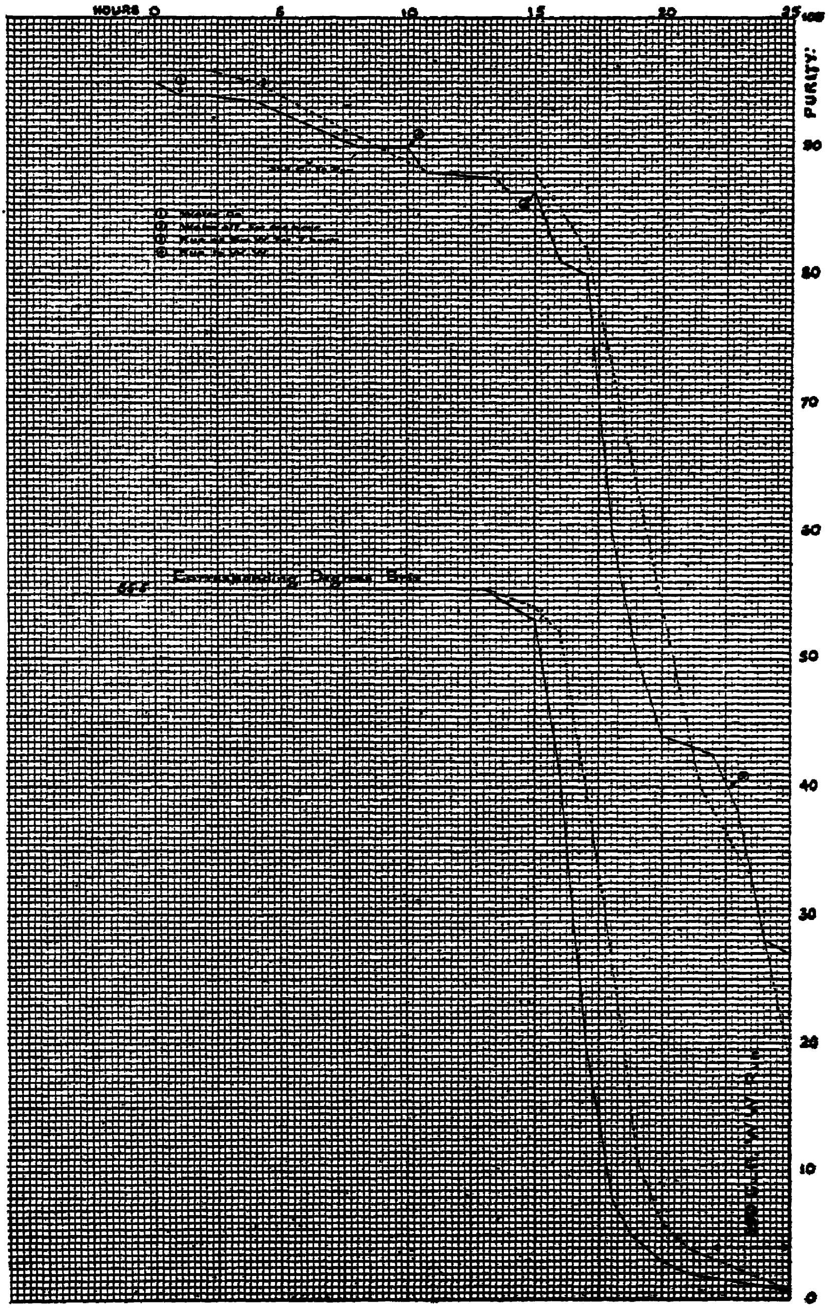

HOURS 0
5
10
15
20
25
100
PURITY:
90
80
70
60
50
40
30
20
10
0
Corresponding Degrees Brix
W. W. Run

Diagram XII.

volume run between the second and eighth hours. Note also that at the thirteenth hour the first decline of Brix is to be remarked. From the fifteenth hour and onward the fall of Brix and purity are coincident and approximatelly parallel, illustrating this important fact—to be observed in all cases—that the moment of dilution of the liquor upon the char surfaces takes place, some of the adsorbed non-sucrose substances are washed into the diluted solution and thus carried back into the refinery by way of the sweet-waters. The necessity of watching this point (3) will be apparent if liquor not lower than a certain purity is to be gathered.

Again, without measuring the sweet-water, a fair approximation of its volume can be obtained by noting the number of hours a filter runs to sweet-water. In the cases illustrated, the filters had run 7½ hours to sweet-water, therefore 7.5 x 50 would mean that 350 cubic feet of sweet-water has been run from each filter.

In like manner the volumes of waste-waters are fixed, or considered as running at the rate of 200 cubic feet per hour. About 600 cubic feet will therefore represent the minimum from each filter, to which must be added 500 to 600 cubic feet held by the char and pumped out by the air pump. This pump water should, of course, contain very little sucrose and, indeed, general solids, if the sweetening-off has been properly done. Allowing a total minimum of 1,200 cubic feet of waste-water and pump water—both flowing to a common tank—per filter, the data of analysis will provide a means of calculating the loss of sucrose for each filter. For purpose of closer control, actual measurements of the volume of waste-water may from time to time be advisable; and purity and total solids determined.

But in order that a complete view may be had of the char-house operations, from hour to hour, the superintendent of the department is charged with placing on record certain prescribed data, preferably in a tabulated form.

Relative to kilns, the rate of drawing should be recorded, which may most conveniently be done by noting the quantity of wet char from the filters sent upon each drier hopper of the battery A, B and C kilns. The quantity of char so charged to each kiln of the battery should be fairly uniform, implying that the work of revivifying is equally distributed upon all the kilns.

The cycle history of a filter requires more detail and should be tabulated in a manner exhibiting every stage of the cycle. The beginning and duration of each operation should be recorded from the time packing of a filter begins to the hour of the completion of the cycle.

For the convenience of maintaining such a record a properly tabulated black-board is usually placed in the gutter room and the data written in. When a cycle has been completed the record may be rubbed off and the space upon the board will be ready for the next cycle history.

1. Packing.
2. Settling.
3. Liquor on.
4. Syrup on.
5. Refilt. Liquor on.
6. Water on.
7. Sweet-water run.
8. Waste-water run.
9. Pumping.
10. Drawing.

Char Filter Record

B (Date)	1	2	3	4	5	6	7	8	9	10	11	12
PACKING							11 1					
SETTLING							12 3					
LIQUOR ON							15 24					
SYRUP ON							15					
REFILTERING												
WATER ON												
S.W. RUN												
W.W. RUN												
PUMPING												
DRAWING												

Fig. IX.

Black-board records may take many forms, but the one suggested here may serve as illustrative of what may be required. A board, or several, if the filter battery of a section is greater than ten or twelve filters, is drawn on the following lines:—Column "a" is permanent and designates section or grade of the char-filters—in this case B char. In the lower squares, one to ten, are printed in the words as listed above. Column "b" is repeated as many times as there are filters to be recorded on the board. In the sketch number seven represent the number of a filter under record. The squares—1 to 10—are crossed by a diagonal line, and the records are entered above and below the diagonal as the operations indicated in column "a" are being performed. Above the diagonal line is written the time when the operation is begun, below the line the duration of the operation. As instanced in the figure packing began at 11; settling at 12; liquor on at 15 o'clock. The numbers 1 and 3 will be written below the diagonal, showing that the operations required 1 and 3 hours respectively, and so on, the entire cycle history being thus recorded.

From the time that a filter begins to run its filtrate, the flow of the filtered liquor, syrup, sweet-water or waste-water are all directed to the particular gutter provided.

As the purity, color and density of the filtrates change, and in consequence the purpose for which they are to be used changes, these three characters must be under observation and the flow of the filtrate diverted at the proper time so that there shall be as complete a separation of filtrates of diverse qualities as possible. In the chapter on Purification general discussion relative to filtered liquors and syrups has been dealt with in sufficient detail, but the disposition of sweet-waters and waste-waters remains to be considered.

Attention has already been drawn to the rapidly descending purity and density curves in Diagram XII, and it is at this stage of the filter cycle that a precise schedule is to be followed for the diversion of the filtrates to the distributing gutters.

Using the typical curves of the diagram as a means of illustrating the treatment of the filtrates, it may be postulated that the filter in question shall be allowed to deliver liquor of minimum purity of, say, 85. The flow of liquor in this case would be diverted to the sweet-water gutter at the end of the fifteenth hour. But even at 80 purity the liquor may still be of excellent color and in this respect, therefore, serviceable for soft sugar boiling. If, however, the density curve be inspected in correspondence with the 80 purity, it will be seen that the Brix relative to 80 purity may lie between 20 and 30. This is a density of solution to be handled by the effects and not by the vacuum pans, and therefore it would be advisable to run the sweet-water as indicated at 3 in the diagram. Neither must it be overlooked that reduced density means dilution of color.

Again, relative to waste-water, which is shown as beginning at the twenty-fifth hour, it is a question as to whether it is advisable to take a thin liquid of only 40 purity back into the house or send it out of the process as waste-water. At 40 purity the density curve shows the Brix to be in the neighborhood of two degrees. It would therefore be necessary to evaporate something like 56 pounds of water to recover 1.26 pounds of solids of which only 0.5 to 0.6 would be sucrose (See formula, Page 111).

These important points in filtration are to be controlled by the laboratory and the superintendent staff, and the control chemist shall see that the schedule of changes to be made at the filters are strictly adhered to. On general principles filtrates are run as sweet-water when the density falls to 50 Brix, or possibly as low as 45, if the evaporating capacity of the vacuum pans permit of some dilute liquors being sent to them.

A filter should never be sent to waste-water before the purity and density of the filtrate has been determined. If the schedule calls for waste-waters to be run at, say 2 to 3 Brix and between 35 to 40 purity, samples will be taken to the laboratory when the prescribed density has been reached, and if the purity has fallen to the figure required waste-water running may be begun. If, however, the purity should be found too high it would indicate that sweetening-off has not been properly watched. The filter, in this case, would be allowed to rest for 30 to 60 minutes, run again to sweet-water, for 10 to 15 minutes and a second purity test made. A persistent relatively high purity in waste-water is always an indication that packing, settling or sweetening-off have not been performed in a proper manner, and any filter running abnormally high waste-water should be watched to determine the loss of sucrose resulting in such an event. A drip sample should be taken, or a sample tested every 30 minutes. Usually high purity waste-water will not persist for any length of time, but it is well to watch any suspected filters so that assurance may be had upon the actual extent of sucrose loss. Normal sucrose loss in waste-water should never go beyond 200 pounds per filter, and may be found as low as 75 to 80 pounds.

The daily estimate of filter efficiency is to be followed by having "drip samples" taken of the chief liquors and syrups. Drip samples are obtained by continous syphoning off of a small quantity of liquor into a covered vessel, and the so-collected sample tested for purity and reaction. The reactions of the A and B liquors should vary but slightly, if proper control in this respect has been maintained in the affination work, and the prescribed alkalinity has been established in the raw liquors and syrups as they issue from the presses, uniform acidity will, as a matter of course, be observed in the char-filtrates. Under Chemical Tests and Liquor Reactions, page 170, the limits of the acid reaction of the char-house filtrates are given, and it but remains to say, that a slight acidity should never be exceeded, since an acidity of 10° to 20° is quite sufficient to maintain the acid reaction desirable during vacuum pan boiling.

In conclusion, and referring once more to the question of sweet-waters and waste-waters, too much stress cannot be laid upon the necessity of keeping the control chemist well in touch with this phase of the filter work. Under all circumstances when a filter has reached the sweet-water stage, it should never be allowed to flow beyond a prescribed limit, and if there be any suspicion that sweetening-off is not following a normal course, the particular filter should be taken especially in hand and tests of density and purity made of the filtrate until normal conditions are again approached. If the rate of flow has been too great, for instance, it will be observed that both the density and purity curves as illustrated in the diagram, will flatten at 30° to 40° Brix instead of at 10°, as shown in the diagram. This condition will move forward to the point when the density has fallen to a degree when the issuing sweet-water must be changed to waste-water, but as the purity curve will have flattened in sympathy with the density curve, a waste-water of 40 to 50 purity may have to be sacrificed or run back into the house, necessitating extra expense in the evaporation of an abnormal quantity of sweet-water.

It is therefore always advisable to provide time for "resting" a filter on sweet-water, *viz.*, at sometime between points 3 and 4 of the diagram; the water should be shut off at the *head* of the filter and the running taps opened to flow 150 to 200 cubic feet per hour. The pressure in the filter will cause this outflow for 10 to 15 minutes and in the meanwhile any accumulated "gas" in the filter will have collected at the top and may be drawn off by means of a gas-cock provided for the purpose. After such treatment sweetening-off may be resumed with good effect.

The point at which waste-water shall be run is determined by the purity of the last stages of sweetening-off, and also by calculating the probable cost chargeable to concentrating such thin liquors to say 55° Brix. The formulae for calculating the evaporation have already been given and the cost of this work is finally determined by considering the cost of fuel and efficiency of evaporation. Taking these data and the prevailing value of sugar into consideration, it may, under certain conditions be economic to allow sweet-water to run back to the house at 1° Brix while at other times 2° Brix might without pecuniary loss be sent out as waste-water.

CHAPTER VI

TECHNICAL AND COMMERCIAL BALANCES

Balance sheets should be divided into two distinct heads, *viz.*, Commercial and Technical.

The Commercial Balance should be arranged to display at a glance the financial aspect of the work accomplished, while the Technical Balance, as the name implies, should be so drawn that the technical staff can obtain all the information necessary to follow the work of the individual units of a refinery and judge of the efficiency of these units, also to determine whether any *avoidable* losses of material have occurred, and, if so, where—so that a repetition of the loss can be guarded against.

It is a common practice on this continent to combine with the commercial reports and stock sheets a Technical Balance, drawn up along the lines shown in the appended Statements I and II.

Such balances purport to show the production data as also the working of the refinery and the disposal of material entering it. They do, indeed, make available the facts and figures necessary from which to calculate efficiencies, but scarcely provide a ready method of visualizing the true relative efficiency of results obtained.

The two statements representative of work accomplished by two refineries working under different conditions, will illustrate our meaning. They have been drawn up on a percentage basis, eliminating stock on hand by the method shown on page 212.

It is generally conceded that from 0.9 per cent to 1.25 per cent loss of sucrose upon sucrose entering, according to working conditions, is a fair unavoidable and unaccountable loss. In fact, no such limits can be placed upon any refinery, unless it always runs along standard lines, and a fair loss be predetermined. Statement I shows work done in a refinery where the bulk of the production was in the form of hard sugars, and the unaccountable sucrose loss appears remarkably small. Compared with it, the Statement II, in which the productions include

STATEMENT I

	Weight	Pure	I. S.	Moist	Ash	S. M.	Not	Pol.	I. S.	H_2O	Ash	S. M.	Not
Raw entering	100,000	96,640	600	780	660	200	1,120	96.64	0.60	0.78	0.66	0.20	1.12
Produced													
Hard sugars	92,696	92,696						100.00					
Soft sugars	1,237	1,114	29	43	27		23	90.05	2.38	3.50	2.18		1.89
Black strap	5,740	2,220	767	1,318	684		751	38.68	13.36	22.96	11.12		13.07
Total	99,673	96,030	796	1,361	711		774						
Loss or gain	— 327	— 610	+ 196	+ 581	+ 51	— 200	— 346	— 0.63	+ 32.66	+ 74.48	+ 7.72	— 100.00	— 30.89

STATEMENT II

	Weight	Pure	I. S.	Moist	Ash	S. M.	Not	Pol.	I. S.	H_2O	Ash	S. M.	Not
Raw entering	100,000	95,790	1,170	1,140	580	170	1,150	95.79	1.17	1.14	0.58	0.17	1.15
Produced													
Hard sugars	75,206	75,206						100.00					
Soft sugars	19,840	17,622	782	851	224		361	88.82	3.94	4.29	1.13		1.82
Table syrups	574	183	218	112	24		37	31.78	38.06	19.50	4.14		6.52
Black strap	3,110	1,149	615	501	269		576	36.93	19.79	16.11	8.66		18.51
Total	98,730	94,160	1,615	1,464	517		974						
Loss or gain	— 1,270	— 1,630	+ 445	+ 324	— 63	— 170	— 176	— 1.70	+ 38.03	+ 28.42	— 10.86	— 100.00	— 15.30

NOTE The high percentage increase of invert sugar in Statement II is due to artificial inversion in the production of table syrup; correcting for this the loss of sucrose becomes 1.5 per cent.

a large percentage of soft sugars and some table syrup, shows what appears to be a heavy sucrose loss.

But let us analyse the two statements from a commercial point of view. To do this we must place an arbitrary value on the various products.

Let it then be assumed that the sucrose in the raw products entering is worth, say, 10 cents a pound, all hard sugars 10 cents a pound, soft sugars 9.5 cents a pound, table syrup 5 cents and black strap 0.5 cent. We then have according to Statement I:

Sucrose entering:

96,640 pounds at 10 cents $9,664.00

Produced

Hard sugar	92,696 lbs. at 10¢	$9,269.60
Soft sugar	1,237 lbs. at 9.5¢	117.51
Black strap	5,740 lbs. at 0.5¢	28.70
		$9,415.11

P = Per cent yield in dollars.

$$\frac{9,415.11 \times 100}{9,664} = 97.42\ \text{P}$$

According to Statement II:

Sucrose entering:

95,790 pounds at 10 cents $9,579.00

Produced

Hard sugar	75,206 lbs. at 10¢	$7,520.60
Soft sugar	19,840 lbs. at 9.5¢	1,884.80
Table syrup	574 lbs. at 5.0¢	28.70
Black strap	3,110 lbs. at 0.5¢	15.55
		$9,449.65

P = Per cent yield in dollars.

$$\frac{9,449.65 \times 100}{9,575} = 98.65.$$

Presenting the problem from another point of view, let it be assumed that the cost of the raw sugar in Statement I be 7 cents and that of Statement II, 6.94 cents and the refining cost respectively 1.5 cents and 1.55 cents, the commercial balance on this basis will still be in favor of Statement II, thus:

Cost of raw + refining cost	
100,000 at 7 cents + 1.50 cents	$8,500.00
9,415.11 — 8,500 = 915.11.	
100,000 at 6.94 cents + 1.55	$8,490.00.
9,449.65 — 8,490.00 = 959.65.	

Thus from a financial standpoint Statement II shows a far more satisfactory recovery than is shown in Statement I.

There are various ways of displaying such facts, but it is not within the scope of this work to discuss the form any commercial statement shall take, as that will depend largely upon the general system of book-keeping of the establishment, and the detail of the information sought. It should not, however, be overlooked that when special products are required there is always likely to be an increased disappearance of sucrose, by inversion or otherwise, and this figure alone, as shown in the usual form of a technical balance, does not necessarily indicate the commercial value of the work done.

For commercial purposes the intrinsic value of productions must be considered in connection with the cost of production. It would be found that, working in each case, under the most advantageous conditions relative to the capacity of a refinery, the cost of running a refinery under conditions depicted in Statement I would be less than as in Statement II, but not proportionately so. The greater profit lies in producing a larger proportion of soft sugars and kindred lines, but the profitable manufacture of soft sugar is provisional upon market demands, and all such considerations are purely financial.

Let us now discuss the technical phase of balances.

The technologist has to be guided in the matter of production by market conditions, nor can he always choose the grade of raw sugar from which he is required to make the products in demand. Thus it can quite conceivably happen that there is a demand for soft sugars at a time when none but high-grade sugars are available, and conversely, the demand for soft sugars may drop off at a time when only low-grade raw sugars, probably containing a high percentage of invert sugar, are on hand, thus necessitating the production of an unusually large percentage of black strap (see Specific Refining Yield, Part I, Chapter II). In such a case the technologist must simply make the best of conditions over which he has no control, and it is with the purpose of showing whether or not this has been done that the technical balance is drawn up. The object is to show, not what *might* have been done with a given raw, but whether it was efficiently handled under the existing conditions, and in this respect the following points have to be considered:

1. The quality of the raw sugar as indicated by the analysis, and its potential yield of the products required.
2. The percentage yield of actual productions and their composition.
3. The percentage losses or gains of the component parts of the raw material entering, which, for good work should compare closely to the percentage shown under valuation of the raw sugar refined.

THE BASIS OF A TECHNICAL BALANCE SHEET

In order that such a balance sheet may be compared with a preceding or subsequent one, it is necessary to classify both raw and refined material, while by-products, or rather residues from period to period are to be standardized.

Raw Sugars.—Raw sugars entering the refinery may be classified as centrifugal, muscavados and molasses sugars, and when a quantity of two or more of these sugars have to be refined simultaneously the average composition of them is computed and taken as the raw material. On the whole, the complexion of the sheets is to be judged by the indication of this average as influencing the refined products and to some extent to be related to the quantitative disappearance of some of the components of this average raw sugar.

Refined Products.—Relative to refined products, the balance sheet is not to be encumbered with the tabulation of the many grades that may have been produced. For instance, as far as technical results are concerned, it is a matter of indifference whether much or little of certain forms of granulated have been produced; whether fancy articles have been made above or below the usual quantity. Such variation does not materially affect the refinery losses; it may increase the total cost of production, etc., but only in a hardly appreciable manner may there be attributable to this fact a slight sucrose loss or increase of invert sugar. Again, the varying amounts and grades of softs are to be averaged, and finally the end-products, such as table syrups and black strap will be placed as separate items. But after every period of refining there remains in the house a quantity of residual material, such as scrap sugar and concentrated sweet-water, etc. This residual material is generally known as "Stock," "Stock in House" or "Stock in Process," and becomes a separate item on the balance sheet. Something of this later.

ILLUSTRATIONS OF TECHNICAL BALANCES

Advancing from simple to more evolved forms of technical balance sheets, a progressive illustration will best indicate the successive steps which a satisfactory form of balance may be arrived at.

Let it be assumed that a balance has to be made upon a raw sugar melt of 40,000,000 pounds, or 20,000 short tons, as a more convenient unit for our immediate purpose. Eighteen thousand tons of this raw may be considered as having been centrifugal and 2,000 tons muscovado sugar. Added to this there are 200 tons of stock in house from a preceding run. We get, then, from the data of analyses of raws and stock:

RAW SUGAR AND STOCK REFINED.

Qual.	Tons	Pol.	R. S.	Ash	Susp.	N. S.	Water
Centrifugal	18,000	96.0	1.00	0.56	0.20	1.24	1.00
Muscovado	2,000	93.0	1.80	0.80	0.28	2.10	2.02
Average	20,000	95.7	1.08	0.58	0.22	1.32	1.10
Stock (A)	200	64.0	8.00	3.00	—	5.00	20.00

"A," stock entering from preceding refining period.

At the end of the run it is found that there has been produced —classified as granulated, softs and stock— as follows:

Granulated	14,140 tons
Softs	4,040 tons
Stock	1,995 tons
Total	20,175 tons

20,200 — 20,175 = 25 tons material lost.

The composition of the refined products is found to be:

	Pol.	R. S.	Ash	N. S.	Susp.	Water
Granulated	100.00					
Softs	89.00	3.25	1.25	2.50	—	4.00
Stock (B)	67.24	5.17	2.53	5.06	—	20.00

"B," stock produced during the refining period. Again, production data shown that from every 100 tons of raw material refined there has been produced.

Granulated	70 tons
Softs	20 tons
Stock	9.875 tons

The components of these products centesimally divided, according to analysis as given above are:

		Suc.	R. S.	Ash	N. S.	Water
Granulated	70 tons	70.00				
Softs	20 tons	17.80	0.65	0.25	0.50	0.80
Stock	9.875 tons	6.64	0.51	0.25	0.50	1.97
		94.44	1.16	0.50	1.00	2.77

But 100 tons of raw material contained:

Suc.	R. S.	Ash	N. S.	Sus.	Water
95.40	1.15	0.60	1.35	0.22	1.28

and, therefore, if from this be taken the centesimal components of the products refined we will obtain the centesimal loss or gain of each component of the raw material worked, thus:

	Suc.	R. S.	Ash	N. S.	Sus.	Water
Raw	95.40	1.15	0.60	1.35	0.22	1.38
Produced	94.44	1.16	0.50	1.00	——	1.97
Loss or gain	— 0.96	+ 0.01	— 0.10	— 0.35	— 0.22	+ 0.59

As far as this balance has gone it is complete in this, that it shows the losses and gains experienced during refining. But as the balance stands there is a variable to be observed that offers difficulty in establishing the value of A as against B tabulated as Stock. Note first the difference in composition:

	Suc.	R. S.	Ash	N. S.	Water
Stock A	64.00	8.00	3.00	5.00	20.00
Stock B	67.24	5.17	2.33	5.06	20.00

The purity of A is 80; the purity of B is 84, and the quantity of B is nearly ten times as great as A. Although in actual practice it is not usual to end a refining period with a stock in process as high as 4,000,000 pounds, nor a purity as high as 84, the illustration as it stands serves to accentuate a point to be dealt with in the succeeding paragraph.

Standardization of Stock in House.—Taking the purity as a basis of value, stock B would be worth, pound for pound, about 5 per cent more than Stock A. But it is quite possible that stock B might be brought to a purity of 74, or even lower, and then its value would be 7 to 8 per cent lower than A instead of 5 per cent higher.

The standardization, then, of stock becomes desirable for the technical, as well as for the commercial balance. It becomes a question of converting the variable stock into its equivalent of a refined article, for stock in process is virtually a semi-refined product.

But two of the least variable outputs of the house are granulated and final molasses or black strap. Furthermore, sugar in the form of granulated has a standard composition, and stock molasses, of any regularly conceived components, will likewise approximate to a uniform composition. If then any particular stock in process be calculated to its equivalent in granulated and, let us say, residual molasses, upon the qualities and quantities thus calculated a definite value may be set as prevailing market conditions may justify. As regards the application of this standardized stock to technical purposes an equally convenient object is served.

Reverting to the stock worked under raw material A, it would be quite in order to extract from it a further quantity of granulated and leave a residual molasses of, say, between 40 and 50 purity. Either one of these figures, or any intermediate one may be taken as a constant; but 50 true purity is convenient and generally accepted.

So the question resolves itself into this: How much granulated and how much stock molasses, at 50 purity, can be extracted from a given quantity and quality of stock in house? Taking stock A as an example, a molasses of 50 purity contains, as this purity indicates, 50 pounds of sucrose and 50 pounds non-sucrose together with a quantity of water. Let 25 per cent be the calculated amount of water and accepted as standard in the molasses.

The percentage sum of non-sucrose (in stock A) is 16, *viz.*, R. S., 8.00; Ash, 3.00; N. S., 5.00. According to the scheme of standardization sixteen parts of sucrose will be associated with this, therefore, thirty-two parts of solids will be associated with ten and sixty-six hundredths parts of water to form the standard molasses, since 25 per cent water is one-third of the solids in the molasses, thus:

$$\frac{32}{2} + 32 = 46.66.$$

The stock A, therefore, may be regarded as yielding on this basis $64 - 16 = 48$ per cent granulated and $16 + 16 + 10.66 = 42.66$ per cent stock molasses. The stock A, therefore, contains potentially

$$200 \times \frac{48.00}{100} = 96 \text{ tons granulated}$$

and

$$200 \times \frac{46.66}{100} = 85.32 \text{ tons molasses.}$$

If stock B be calculated along similar lines it will be found that it contains:

Potential granulated	1,085 tons.
Potential molasses	680 tons.

It is now possible to set the value of stock A against stock B, since the stock molasses of A and B is approximately of the same composition.

But if 73 tons granulated had been made from the melt instead of only 70, the stock B would, of course, have been reduced to

74 purity and the potential granulated to a smaller quantity. In actual practice, however, a standardized molasses will not vary at all in purity, and in reducing sugars, ash and non-sucrose but slightly, so that a molasses from one run may quantitatively be compared with that of any other run without further ado.

But supplementing the simple balance sheet suggested in the preceding paragraphs, it is desirable to construct a more comprehensive one that may be more readily analyzed and reveal the actual and potential products obtained as coming exclusively from the melt of raw sugars.

A table, then, may be constructed embracing only the main quantities involved but at the same time making more prominent the technical achievements of a particular run.

A MORE COMPREHENSIVE TABULATION OF A TECHNICAL BALANCE

Again taking the former quantities of raws and from these 20,000 tons producing 73 per cent tons of granulated and 20 per cent tons of softs, we will obtain as an end product a stock molasses of 74 purity.

The results may be studied in the appended tabulations X and XI.

The illustrative Table X is divided into six parts; in part (a) are ranged the quantities of all materials that have entered into refining operations during a period of work. The weights of materials and the weights of the components are set in a series of columns, while the percentage composition—in part (b)—is set in an adjacent series.

Part (c) treats of the total refined products along lines indicated in (a), so that if the totals obtained in (c) be taken from those in (a), loss and gain will be shown. Part (d), relative to refined products, is the counterpart of part (b).

In part (e) the potentials of stock A and B have been segregated and the compositions of Stock molasses A and B may be compared, and are standardized as already indicated. Here part (f) again takes an analogous position to parts (b) and (d).

TABLE X—WORKED—PART (a)

	Weight	Sucrose	R. S.	Ash	N. S.	Susp.	Water
Centrifs.	18,000	17,280	180.0	100.8	223.2	36.0	180.0
Muscovados	2,000	1,860	36.0	16.0	42.0	5.6	40.4
Total raws	20,000	19,140	216.0	116.8	265.2	41.6	220.4
Stock	200	128	16.0	6.0	10.0	—	40.0
Total melt	20,200	19,268	232.0	122.8	275.2	41.6	260.4
Loss or gain	——	204	2.0	22.0	72.7	41.6	145.0
ANALYSES—PART (b)							
Centrifs.	——	96.00	1.00	0.56	1.24	0.20	1.00
Muscovados	——	93.00	1.80	0.80	2.10	0.28	2.02
Average	——	95.70	1.08	0.58	1.33	0.21	1.10
Stock molasses A	——	64.00	8.00	3.00	5.00	—	20.00
Average	——	95.38	1.15	0.61	1.36	0.21	1.78
ACTUAL PRODUCTION—PART (c)							
Granulated	14,746.0	14,746.0	—	—	—	—	—
Softs	4,040.0	3,595.6	131.3	50.5	101.0	—	161.6
Total	18,786.0	18,341.6	131.3	50.5	101.0	—	161.6
Stock B	1,221.1	722.4	102.7	50.3	101.5	—	244.2
Total products	20,007.1	19,064.0	234.0	100.8	202.5	—	405.8
ANALYSIS—PART (d)							
Granulated		100.00	—	—	—	—	—
Softs		89.00	3.25	1.25	2.50	—	4.00
Avg. sugars		97.63	0.70	0.27	0.54	—	0.86
Stock B		59.17	8.41	4.11	8.31	—	20.00
Average		95.30	1.17	0.50	1.01	—	2.02
POTENTIAL PRODUCTS—PART (e)							
Stock A							
Granulated	96.00	96.00	—	—	—	—	—
Molasses	85.33	32.00	16.0	5.0	10.0	—	21.33
Stock B							
Granulated	467.90	467.90	—	—	—	—	—
Molasses	678.66	254.50	102.7	50.3	101.5	—	169.66
ANALYSES OF POTENTIAL STOCKS—PART (f)							
Stock A							
Granulated		100.00	—	—	—	—	—
Molasses		37.50	18.75	7.03	11.72	—	25.00
Stock B							
Granulated		100.00	—	—	—	—	—
Molasses		37.50	15.13	7.41	14.96	—	25.00

TABLE XI—RAW SUGAR WORKED—PART (a)

	Tons	Per cent
Centrifs.	18,000	90.0
Muscos.	2,000	10.0
Total	20,000	

REFINED SUGARS—PART (b)

Granulated	14,746.0[4]
Gran. in B - A	371.9[5]
Total	15,117.9
Softs	4,040.0
Total	19,157.9[6]

STOCKS A AND B—PART (c)

		Tons
A	Granulated	96.00
	Molasses	85.33
B	Granulated	467.90
	Molasses	678.66

B — A = 678.9 — 85.33 = 593.33

593.33 = 2.97 per cent on raw.

ANALYSIS OF—PART (d) PRODUCTS

	Suc.	R. S.	Ash	N. S.	Susp.	Water
Refd. sugars	97.70	0.68	0.26	0.53	—	0.84
Stock molasses	37.50	15.13	7.41	14.96	—	25.00

PER CENT TONS ON RAW CONTAINED IN—PART (e)

Sugars	95.79	93.57	0.65	0.25	0.51	—	0.81
St. mol.	2.97	1.11	0.45	0.22	0.45	—	0.74
Total	98.76	94.68	1.10	0.47	0.96	—	1.55

THE BALANCE—PART (f)

	Weight	Suc.	R. S.	Ash	N. S.	Susp.	Water
Raw sugars	100.00	95.70	1.08	0.58	1.33	0.21	1.10
Refined products	98.76	94.68	1.10	0.47	0.96	—	1.55
Loss or gain	1.24	1.02	0.02	0.11	0.37	0.21	0.45

YIELDS—PART (g)

	Potential on refined products Per cent	Potential on 100 raw sugar Per cent
Granulated	96.54	75.59
Softs	20.46	20.20
St. molasses	3.00	2.96
	100.00	98.75
Loss		1.25

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[4] Actual production.

[5] Potential gran., *viz.*, B — A = 467.9 — 96 = 371.9; Table XI, Part (e).

[6] 19,157.9 = 95.79 per cent on raw.

EXPLANATORY OF TABLE XI

Upon examination of Table XI it will at once be seen that it draws its data from Table X, and, indeed, it is always advisable to complete Table X before attempting the construction of Table XI.

As already briefly suggested, the special purpose of Table XI is to present, with as few figures as possible, the production obtained from and exclusively attributable to the raw sugars melted, irrespective to the quantity and quality of the stock in house that may have entered with the raws. The form of these data is desirable for two reasons: In the first place, by the elimination of the influence of the stock A upon the refined products obtained, there is removed a variable which such a stock represents in each refining period. Upon final analysis it will appear that such a stock, although standardized, is a *variable as a whole.* In the second place, having the loss or gain of the components shown, as in Table X, there is also presented in the potential yield of the refined products, the true refining value of the raw sugar melted. This is the more a fact in this—though at first sight it may seem strange—that the potential granulated and molasses of stock B, considered and entered as production, can in no sense be a variable, since, by the elimination of stock A, stock B becomes a part of the standard products of any refining period. Therefore we obviously find in Table XI the most direct expression of quantity and quality of the refined products directly obtained from the raw melted.

In the seven parts into which Table XI is divided, are to be found the interrelated factors of a refining period. Part (a) needs no comment, as it is nothing more than a list of the raw sugars melted; the average composition of these is given in the first line of part (f). Of the five items of part (b), two only need explanation, *viz.*, the potential granulated, B—A, and the total, or sum of the first four items, *viz.*, Granulated; Granulated in B—A (potential granulated) and Softs.

Besides the actual production of granulated and softs, *i. e.*, those sugars which have been packed and are ready for shipment, there is to be accounted for the potential granulated that could be obtained from stock B. But contributive to the potential granulated in B and in the granulated of actual production, there are 96 tons derived from stock A which is to be eliminated. Therefore this 96 tons must be taken from the actual or potential granulated shown. Preferably it is to be taken from B, as then the actual production is left intact. Hence B—A, in the case, 467.9 — 96.0 = 371.9, which is to be added to 14,746 tons. The items total to 19,157.9 tons, which is the potential yield in refined sugars obtained from 20,000 tons of raws worked; 19,157 is 95.79 per cent of 20,000.

Part (c) is simply the tabulated standardized stock A and B in tons, of which 593.33 tons is equal to 2.97 per cent of the raw. This figure is obtained by taking molasses stock A from the molasses stock B, thus, in an analogous manner obtaining the weight of the potential molasses solely attributable to the raws.

Conceivably, upon occasion, the quantity of standard granulated and molasses stock of A may be greater than B. Then B—A would be a minus quantity. In such a case all figures relative to molasses and granulated stock would be carried forward as minus quantities in all parts of the calculations.

If from the data of Table XI—parts (b) and (c)—and from the actual productions shown in Table X, part (c), the compositions of the potential products be calculated, they will be found as recorded in part (d), Table XI. In part (e) the component yield is to be found by multiplying the yield 95.79 per cent by the components or analysis given in (d); thus, $\frac{95.79 \times 97.70}{100} =$ 93.59, etc. The sum of the yield of sugars and the stock molasses will therefore give centesimal yield of all of the refined products. If from the average analysis of the raws there be taken the total components of part (e), the remainder will indicate the loss or gain of each component of the raw sugar refined.

Finally under the yield, part (g), a summary of production permits of instant visualization of the relative quantities of refined articles made, irrespective of the actual quantity worked within the period covered by the balance sheet.

As the object of these illustrative balances is to show the method in its simplest form, table syrups and black straps were not considered as produced. If, however, these should have been made, they would not have been incorporated with the refined sugar as refined products, but itemized separately, and would appear in the lists of parts (b), (d), (e), (f) and (g) of the table.

COMMENTS ON THE BALANCE SHEETS

The advantage of supplementing Table X by Table XI lies in the fact that in the latter the data are presented almost exclusively on a centesimal basis and are thereby more easily criticized and compared with the balances, similarly drawn, of earlier working periods. For technical purposes the actual quantity upon which a balance may be made has comparatively little significance, and therefore the number of tons or pounds refined play a secondary rôle only. For instance, the loss and gain shown in the last line of part (f), Table XI, may be compared at a glance with the same line of some previous sheet. Further, in the example given it is to be seen that only 2 per cent invert sugar has been produced; that 19 per cent of ash and 30 per cent of non-sugars have disappeared and must have been washed away in the waste-waters. Again, by comparing a number of such balance sheets, differences of loss of sucrose, ash and non-sucrose will be noted, and sometimes, invert sugar may show a loss instead of a gain. By studying a series of balances critically, and taking the percentage production of granulated, softs and stock molasses into consideration, it may be noted that apparently great divergence of filter losses are to be accounted for in the divergence of refining work necessitated by the greater production of granulated in one period over another.

Take this supposition under discussion. If instead of 20 per cent softs, as shown in the illustrative balance, only 10 per cent had been made, a quantity of black strap would have been produced. Black strap is entirely absent in the balance sheets submitted, but the operation of a refinery producing 20 to 25 per cent softs would be quite different from the methods to be followed when 5 to 10 per cent only are to be made. Two salient features would be observed in the latter case. First, 2 to 4 per cent of black strap would be produced and second, less char-filters, presses, etc., would be needed as compared with those necessary for producing 20 to 25 per cent softs. Why? High percentage of granulated production means higher black-strap production also. But when black strap is to be made, the advantage accruing from this will be to remove the components of it out of the house as soon as possible. This being done, it may appear that 75 per cent or more of the non-sucrose substances never reach the char-filters, etc., a great percentage of the impurities of the raws passing out of the general operations by way of the black-strap magma.

Under such conditions the necessity of char-filtration will be reduced in proportion to the diminished quantity of non-sucrose sent upon the filters. Obviously 70, 80, even 90 per cent of the coloring matter and general impurities of the raws will be found in the black strap produced.

But such differences in refining are at once reflected in the balance sheet following such a period. Less char-filters used means less pure sugar loss; it also means that little ash and non-sucrose have entered the filters and therefore, instead of showing a filter loss—more properly char-adsorption—of ash and non-sugars of from 15 to 20 and 25 to 35 per cent respectively, such adsorption may fall to 5 per cent in one and 10 per cent in the other.

At the other extreme, high production of softs means more sucrose loss since more char-filtering will be needed to obtain the color reduction necessary, and in consequence the eventual balance sheet takes on a different complexion.

By far the greatest loss of raw material takes place through the char-filters; losses in other stations of a refinery may frequently be fortuitous, but filter-loss is inevitable and can be held to normal solely by strict control and supervision. On the whole, it may be taken for granted that under usual refining work 80 per cent of material loss may be charged to char-filtration, and it is no doubt due to this fact that losses collectively are referred to as filter-losses.

APPENDIX

In 1911 and 1912 a decolorant black, "Eponite"—now known as "Norit"—was suggested as a possible decolorizing carbon for sugar solutions, and investigations with a view of ascertaining its value in this respect, necessitated the adoption of some method of color determination in the different sugar solutions upon which experiments were being made.

Hence the adoption of the Lovibond instrument for this purpose, and the standardization of color analysis as indicated in the article.

Since 1914 many "activated" blacks have been put upon the market, and the Hess-Ives tintometer introduced and variously applied in the investigation of both quantitative and qualitative estimation of activated carbons.

The relative merits of the Hess-Ives and the Lovibond tintometers need not be discussed here as many articles have appared since 1917 in different chemical journals (American and British), nearly all publications especially dealing with sugar, its manufacture and refining. To these the reader is referred; a partial list of which will be found below.

As regards the data obtainable from the use of the Lovibond Tintometer, the authors are of the opinion that with due precautions this comparatively simple apparatus can be made to yield valuable practical data in all tintometric work upon sugar solutions. More particularly is this made possible by a novel adaptation of the Lovibond principle, recently applied to color work.

The Lovibond tintometer was modified by us in the following manner:

1. Instead of the usual open-view trough, two tubes are led down to openings, one immediately above the cell for the ex-

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[1] *Industrial and Engineering Chemistry* (American Chemical Society); "A New Colorimeter," June, 1916, page 517; "A Numerical Expression for Color as Given by the Ives Tint Photometer," August, 1916, page 711, also March, 1917, page 282; "Testing Decolorizing Carbons," May, 1924, page 498; "Comparative Ash Adsorption of Vegetable and Bone Chars," February, 1924, page 165; "The Influence of Variation in Concentration on the Adsorption Spectra of Dye Solutions," January, 1924, page 35; *Journal of the Society of Chemical Industry*, 1906, page 552; *International Sugar Journal*, 1917, pages 278 and 306.

amination of the solution for analysis, and the other above the rack for the tint slides. The ends of the tubes are firmly secured by metal plates through which they extend at the upper end for a short distance. Over these tubes by means of caps is fitted a box, containing a pair of Fresnel's rhombs. and centered over the junction of the rhombs is a viewing telescope. By this means the divided circle displays the colors of the solution and the standard tints in juxtaposition, enabling the operator to make an accurate match of the tints.

2. The whole apparatus is rigidly fixed at a predetermined angle assuring a constant angle of reflection, it being merely necessary, for accurate work, to direct the light on to the reflecting plate in such a manner that the angle of incidence is always the same—a simple matter in diffused light or where artificial light is used as the source of illumination.

3. Finally a hinged cover is placed over the cell and slides to prevent any light entering the instrument except that which passes through the solution and the tint glasses.

The accompanying sketch, Fig. X, will convey a clear concept of the apparatus as modified by us.

In conclusion it may be remarked that the quantity of color in a raw sugar solution is so small that it is practically unweighable, although the *intensity* of the color may be very pronounced. In all colorimetric work, therefore, it is necessary to make *volumetric* comparisons of color intensities, by working on sugar solutions of a fixed density, and observing them through a constant standard cell.

The results obtained should be recorded to show the density of this solution and the size of the cell used. We have found it practicable in all cases to use a 1-inch cell and have divided the various solutions into three classes, *viz.*

1. Char filtered liquors, granulated and refined syrups to be observed at the average normal density—55° Brix.

2. Raw sugars, char-filtered low-grade syrups, sweet-waters, etc., at 10° Brix.

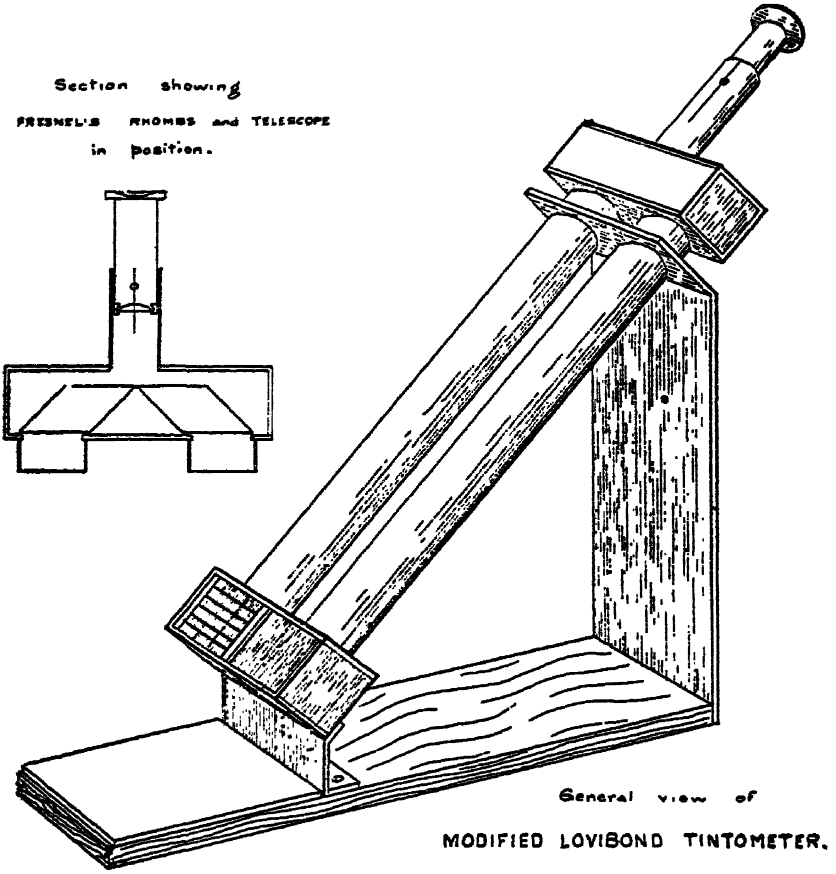

Fig. X.

3. Thin final sweet-waters, all dark syrups and molasses at about 2° Brix, or such further dilution as may be necessary, making all color comparisons at the same density, etc. Where it is desired to calculate the percentage color "removed" any convenient method may be adopted, such as bringing all observations to a 100 basis by multiplication of the Brix, thus: $10 \times 10 = 100$; $55 \times 1.82 = 100$; $2 \times 50 = 100$. This does not represent the *actual* work done, but affords a fair comparison as long as all determinations are made under identical conditions.

Example of Experimental Filtration:

	Grams, solids	T. observed at	Total color
Liq. entering at 55° Brix (hot) 7,200 cc.	3,960	6.50 at 10° Brix	468,000
Filtered liq. at 55° Brix (hot) 6,650 cc.	3,652.5	4.76 at 55° Brix	6,650 × 4.76 × 1.82 = 5,631
Sweet-water 10° Brix 1,050 cc.	105.0	0.85 at 10° Brix	1,050 × 0.85 × 10 = 8,925
Waste-water 2.5° Brix 3,500 cc.	87.5	1.46 at 2.5° Brix	3,500 × 1.46 × 40 = 204,400
	Solids recovered		Total color accounted for
	3,845.0		218,956

From these figures we may calculate that from the filtrate as liquor 6,650 × 6.5 × 10 = 432,250

$$\frac{(432.250 - 5,631)100}{432,250} 98.7 \text{ per cent original color removed.}$$

Similarly we may calculate the color and solids retained by the char, etc.

TINTOMETRY[8]

The appended article (first published in *Sugar*, August, 1917) is a resumé of a method developed for color analyses by the aid of the Lovibond Tintometer.

That the possibility of placing on record the colors of various commercial products in some readily available form, "Is a consummation devoutly to be wished," will be admitted by any one who has been dealing with products in which the color element plays a more or less important part. Not only is it desirable to have such a record expressed in some suitable form, but the convenience of being able at any time to readily *reproduce* the color for inspection and comparison is another factor of immense value, and would enable any one to definitely follow many important results involved in the production of some standard colored article, or be an aid in determining what tints in a raw material, sugar for example, might be expected to require special attention for removal or modification. As the data to be submitted will almost exclusively refer to tintometric measurements of sugar solutions, a word or two on the growing importance of color analysis of raw sugars will not be out of place.

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[8] By C. F. Bardorf, in *Sugar*, 1917.

In the early days of sugar refining the color of raw sugars were judged by the experienced eye. Sometimes experimental filtrations were made over a standard bone-black (each refiner had his own standard of course)and so some sort of insight was obtained of the probable behavior of the particular sugar under actual refinery operations. Also in the line of general experimental work the determination of color values was always a problem, and the various colorimeters, although serviceable, fall short in many desirable respects, more especially in this, that they can give no *analysis* of the color, nor is it possible to readily reproduce the color so determined. Four of the five types of colorimeters, the Payen, Bentzke, Duboscq and the Stammer instruments, are based upon measuring the quantity of color by observing a column of liquid by means of a varying depth of the liquid and calculating from this a comparative color value. The Salleron colorimeter endeavors to attain the same object by measuring the dilutions of a specific volume of liquid observed through a constant depth. It will be quite apparent, when we come to examine the results obtained by means of a tintometer, which in a higher degree is also a colorimeter, that for most important work, all the older types are utterly inadequate. They give comparative quantitative results, but qualitative estimation is practically out of the question with them.

With the aid of the Lovibond tintometer, however, very reliable color analysis can be made as well as the character determination of any clear-colored liquid. The tintometer is simplicity itself, being little more than an almost rectangular, oblong box with appropriate peepholes and a white plate for reflecting the light. The box can be set to any convenient angle, which, of course, must be kept constant and should be determined upon once for all. The cells for the colored solutions to be examined, have two parallel ends of colorless glass and are furnished for observation through depths of from one-sixteenth to 1 inch of liquid. For determination of tints in lightly colored liquids, natural waters, etc., 2-foot cells are frequently used in an appropriate tintometer. A small rack, placed beside the observation cell holds the colored glasses necessary for each determination. Finally, a set of about 400 small colored glass plates. Yellow (Y), Red (R) and Blue (B), are necessary for a wide field of work. The glasses are accurately graded and are numbered so that if the same number of a yellow, red and blue plate be superimposed they produce a neutral tint or black.

Before describing the methods evolved for the particular purpose of the investigations in hand, it may be well to give a few abstracts from Lovibond's papers, which he from time to time, read before the Society of Chemical Industry, and which will be found in the *Journal.*

He says, "Color standards must be available for use under ordinary daylight conditions, and for determining the color of liquids, solids or gases they may be either fixed points or scales, but scales have a much wider range of application. The character of the light used in preparation must be defined, both for intensity and composition. The angle of incidence of the light must be defined, and also the distance of the eye from the object of vision, the latter on account of intensity of the light reflected, the former as causing variations in color and intensity.

"In order to standardize the sensation of color it must be correlated to a physical color-constant for which the following have been suggested, and used with more or less success. The solar spectrum, the spectrum of the arc light, the colors of defused daylight, pigments, chemical solutions. The solar spectrum naturally suggests itself as a standard, and at first appears to be promising, practical work, discloses so many complications that up to the present no satisfactory results have been arrived at."

After discussing the value of the spectrum, chemical solutions, direct sun light and other sources of light, he comes to the conclusion that, "diffused daylight as an illuminant is free from most, if not all, the disadvantages of direct lights and has the distinct advantage, that while one part of the impinging beam illuminates the object, the other part illuminates the color standard. In reading color sensations for record constancy is obtainable by observing certain conditions all of which appear to be within the limits of definition. The principle points to be defined are intensity of light, the angle of incidence and the degree of diffusion."

In conclusion he states this law: "The color of a pure substance at a *particular density* is constant. Note—This law affords means of checking the standard color scales as by matching them with pure-colored substances of known density any departure from absolute can be at once ascertained.

"Every substance has its own specific light rate of light absorption developing definite color sensations for definite increasing densities."

Malt.—Finally his remarks on the color of malt (extract) are strictly applicable to sugar solutions and accentuate the necessity of distinguishing between "color-character." He writes: "A wide distinction must be made between the terms color-depth and color-character. When we speak of malt as being 'light' or 'dark' in color we only express our idea of the color depth of the sensation without regard to the color-character. It is only when a number of malts similar in color depth but varying in quality are brought together, and the constituents of the color are separated by analysis, that we realize how great a variety of color-character is found under a single expression of color-depth."

In the preliminary steps for the establishing of a standard method of procedure for the analysis of color in sugar solutions, Lovibond's law is to be kept in mind and his investigation on the colors of malt are exceedingly useful. But another fact immediately obtruded itself, and it was this: that with a change of density, or with a change of depth of observation cell, tints change not only in quantity but also in quality, or character, as Lovibond terms it. But such changes are peculiar in this, that they are not proportional to change of depth or density of a solution, *i. e.*, a solution observed through a one-half inch cell gives *less* than half the color quantity observed through a one-inch cell, and the color-character also changes to a marked degree; the percentage of red is usually higher.

To illustrate this, solutions of different concentrations were observed (a) of bichromate of potash in one-half inch and one-quarter inch cells, and (b) tartrazine in one-sixteenth inch cell, as shown in Table XII.

TABLE XII

Sol. of bichromate of potash		½-in. cell	Per cent	¼-in. cell	Per cent
0.09 per cent	Y	20.00	95.5	—	—
	R	0.95	4.5	—	—
0.18 per cent	Y	22.00	96.6	9.00	94.30
	R	0.78	3.4	0.54	6.70
Tartrazine in 1/16-in cells		**Brand W. T.**	**Per cent**	**Brand K**	**Per cent**
0.2 gram in 500 cc. water	Y	18.00	95.7	20.68	99.04
	R	0.80	4.2	0.20	0.96
0.1 gram in 500 cc. water	Y	6.60	93.7	10.00	100.00
	R	0.44	6.2	—	—

Turning now to the specific purpose to which the tintometer is to be applied, the comparison of various solutions of raw sugar and refinery products generally, it was necessary to establish a rigid standard so that one of the most valuable features of tint-recording might be preserved, and that is, the possibility of being able to readily reproduce the color by color-plate building from the records of the color analysis once made.

Naturally a depth of color must be chosen which should meet the large range of color encountered in raw sugars, and at the same time present none of them too dark nor too light for undisputable judgment at any time. It was very soon discovered that the practical limits, for the purpose, lay within defined depths beyond which the color analysis gave dubious results. In some cases the total color being too bright or certain shades or tints difficult to detect, and in some instances, a particular component would actually disappear.

After a number of preliminary trials, it was finally decided to work upon a 10 per cent solution observed through a 1-inch cell. From numerous color analyses it was concluded that this observation standard gives the most satisfactory results, making observation comparatively rapid and accurate, and in no case eliminating any of the tint elements.

The data below will illustrate this and also again indicate the changes of color-character referred to in Table XII.

TABLE XIII

Sugar solutions		1-in. cell	Per cent	½-in. cell	Per cent
20 per cent	Y	34.00	86.4	9.80	80.8
	R	5.00	12.7	2.20	18.1
	B	0.34	0.8	0.13	1.1
		39.34		12.13	
10 per cent	Y	9.80	80.8	3.50	76.0
	R	2.20	18.1	1.10	24.0
	B	0.13	1.1	—	—
		12.13		4.60	
5 per cent	Y	3.50	76.0	1.50	73.5
	R	1.10	24.0	0.54	26.4
	B	—	—	—	—
		4.60		2.04	

Again another question arose: given two solutions of known color composition, will definite mixtures of these yield concordant color composition, determined and calculated?

Experiment proved this not to be the case; the calculated figure always gave a higher color than that determined by actual observation, variation lying between 10 to 40 per cent according to the nature of the mixture.

Two solutions, A and B, were color analysed and gave the following figures shown in Table XIV.

TABLE XIV

		1-in cell	Per cent
Solution A	Y	11.00	68.5
	R	3.40	21.1
	B	1.65	10.3
		16.05	
Solution B	Y	1.65	85.5
	R	0.28	14.5
	B	—	—
		1.93	

From these, two different mixtures were prepared; I—three volumes A and 1 volume B; II—equal volumes of A and B.

		Determined	Per cent	Calculated	Per cent
I. 3 vols. A and 1 vol. B	Y	8.30	71.6	8.66	72.75
	R	2.20	19.2	2.62	19.45
	B	1.05	9.1	1.24	7.70
		11.45		12.52	
II. 1 vol. A and 1 vol. B	Y	4.90	74.8	6.32	77.00
	R	1.35	20.6	1.89	17.00
	B	0.30	4.6	0.82	5.10
		6.55		9.03	

The color in each case determined is lighter than that calculated.

Excessive dilution brought about a remarkable change in the color, both in quantity and character.

A sugar solution reduced from 55° Brix to 10°[3] gave the figures shown in Table XV.

TABLE XV

		1-in cell	Per cent
55° Brix	Y	68.7	46.0
	R	76.5	51.2
	B	4.0	2.6
10° Brix	Y	11.0	71.4
	R	3.8	24.7
	B	0.6	3.8

One more item had to be dealt with before venturing upon the application of the tintometer for refinery control, and that was to find a ready method of obtaining a clear solution for tintometric observation. Since all solutions of raw-sugar products are more or less turbid, and must therefore be clarified, small quantities of diatomaceous earth were first used. But this earth—as also Al cream—has a tendency to absorb colloidal color from the solutions and clarification had, therefore, to be obtained by filtering the solutions through a layer of asbestos, passing the liquid through the filter several times if necessary.

[3] While the reduction of a sugar solution from 55° Brix to 10° Brix is not (owing to contraction) a true volumetric dilution of 1:4.5, the error of dilution will not account for the great difference noted, and the determinations have been made to show the relative differences both in quantitative intensity and quality of color of solution at the two densities of *sugar* solutions at which color observations are usually made.

The comparatively strong decolorizing effect of the earth is well illustrated by the color analysis of a solution before and after being shaken up with about 10 per cent of the earth to solids of the raw sugar solution (see Table XVI).

TABLE XVI

	Color	Raw sugar Per cent	Color	Raw sugar and earth Per cent
Yellow	18.00	84.30	6.60	80.0
Red	3.20	15.00	1.60	20.0
Blue	0.15	0.70	—	—
	21.35		8.20	

About 60 per cent of the color was removed by the earth.

Considering the vagaries and deviations observed in tintometric measurements as indicated above, the practical application of the instrument at first sight appears of very limited value, but most of the difficulties vanish by keeping strictly to the conditions laid down, so that by reasonable precautions, information not otherwise attainable can be pressed into valuable service. Indeed as compared with results obtainable by means of Stammer's colorimeter, the use of the Lovibond is certainly wider and its results more informative.

Coming now to the practical application of tintometric analysis to mill and sugar-house products, the Lovibond may be considered useful relative to color analysis upon I, Raw Sugars; II, Refined Products and for, III, Measurements of Char Efficiency; IV, Experimental Work.

The following illustrations may prove interesting.

RAW SUGARS; COLOR REMOVED IN PROCESS OF REFINING

The consideration of one class of raw sugar will suffice to point out the features of color variation. Take 96 centrifugal crystals as examples.

It will be quite evident from inspection of Table XVII that for unity of raw material refined an appreciably greater quantity of color will have to be removed in some cases over others. Taking two extreme cases (Nos. I and II, Table XVII) there is 60 per cent more color in No. I than in No. II. Also note that in Nos. I and II there appears nearly double the amount of blue over Nos.

III and IV. This fact, in the experience of the author, seems to indicate that more colloidal color has to be dealt with in those sugars showing a relatively high percentage of the blue element in the color constituents.

TABLE XVII

No.	Pol.	I. S.	Ash	Susp.	Non-S	Water
I.	96.2	0.79	0.46	0.35	1.30	0.90
II.	96.0	1.42	0.51	0.20	0.62	1.25
III.	96.2	1.35	0.48	0.24	1.08	0.65
IV.	96.2	1.59	0.52	0.27	0.72	0.70

COLOR ANALYSES.

No.	Y	R	B	T[10]	Per cent Y	Per cent R	Per cent B
I.	62.0	27.0	5.6	94.6	65.5	28.5	5.9
II.	38.0	17.0	3.4	58.4	65.1	29.1	5.8
III.	54.0	18.0	1.5	73.5	73.4	24.5	2.1
IV.	49.0	18.5	2.2	70.7	69.3	27.6	3.1

Applying such data to the measurement of refining results, average samples of all raw sugars and all refined products are to be made and put under tintometric determination, and from the figures so obtained the color removed for a refining period can then be calculated.

Measurement of Efficiency.—For checking the general efficiency of the char-filters and other steps in refining operations the tintometer can be made to correct the fallible, unaided eye-judgment of even the most experienced. Methods can readily be devised to suit the special requirements of any establishment.

Experimental Work.—In no other line of work has the tintometer been more valuable and furnished more interesting data than in experiments more or less directly connected with actual sugar-house operations.

In the first order of importance is the determination of the filtering value of a particular bone-black which may be in the battery of filters, or is about to be used by itself or in conjunction with other working chars. But the great question primarily is: "What is a good char?" Or given two chars, which of the two is the better? Unfortunately there is no single factor of quality, nor several of them combined, to definitely answer this question, indeed, unless one of the commonly recognized qualities of a char is decidedly off normal, it is almost impossible by the data before one to decide in favor of one or the other.

---

[10] T = Total, *viz.*, the sum of Y, R and B.

Comminution, apparent specific gravity and chemical analysis should, of course, be considered and a velvety lustre is always a good characteristic, but one may be led astray even when all three factors look normal. Therefore, experimental filtration is the final and deciding proof of the relative efficiency of a char, and the tintometer picks out differences in char-filtration quite undetectable by the unaided eye.

On occasion three pairs of chars were tried out as to their filtering qualities under absolutely uniform conditions, and similar chars were operated upon simultaneously, *i. e.*, in pairs making three filtrations in paired experimental filters. The entire course of filtration proceeded along standard lines, previously laid out, which had been used on many occasions. Color analyses in these experiments were uniformly made on solutions at 55° Brix observed through a one-fourth inch cell.

The six chars may be designated A1, A2, A3 and S1, S2, S3. The respective numbers indicate that the pairs of char are similar in composition, and filtration was made simultaneously on each pair.

Comparative Filtration of A and S Chars. Basis of Filtration. —Sets of comparative filtrations were made through cylindrical filters, 13 inches by 6 inches, containing 5 kilos of char. Of raw liquor, 7,200 cc. at 55° Brix, equal to 5 kilos of solids were run on to the surface of the char at a rate of 600 cc. per hour. The rate of settling was therefore from 4 to 5 hours according to the apparent gravity of the char.

After all liquor had been run on, water followed at the rate of 300 cc. per hour until 6,500 cc. of the pressed off filtrate had been gathered as liquor, and 1,050 cc. as sweet-water. The speed of water flow through the char was increased to 1,200 cc. per hour and maintained until 3,500 cc. of filtrate had been collected as waste-water.

During passage of the liquor and the first 1,000 cc. of water, the temperature was held at 83° C. and then raised to 85° C. and maintained to the end of the experiment.

TABLE XVIII—CARBON CONTENTS OF THE EXPERIMENTAL CHARS

	Per cent carbon		Per cent carbon
A1	8,25	S1	11.20
A2	10.48	S2	8.02
A3	8.10	S3	9.42

WEIGHT PER CUBIC FOOT

A1	55.5 lbs.	S1	53.0 lbs.
A2	71.9 lbs.	S2	57.9 lbs.
A3	73.0 lbs.	S3	61.3 lbs.

TABLE XIX—COLOR ABSORPTION BY A AND S CHARS

	Original raw liquor	Per cent	6,650 cc. from A1	Per cent	6,650 cc. from S1	Per cent
Yellow	38.5	68.6	1.25	79.6	1.05	81.4
Red	16.0	28.5	0.32	20.4	0.24	18.6
Blue	2.9	—	—	—	—	—
	56.1		1.57		1.29	
Per cent color removed			97.18		97.70	

		A2	Per cent	S2	Per cent
Yellow	38.5	9.60	83.1	2.00	74.6
Red	16.0	1.95	16.9	0.68	25.4
Blue	1.6	—	—	—	—
	56.1	11.55		2.68	
Per cent color removed		97.41		95.22	

		A3	Per cent	S3	Per cent
Yellow	38.5	9.60	84.2	6.20	83.8
Red	16.0	1.70	14.9	1.10	15.0
Blue	1.6	0.10	0.9	0.10	1.2
	56.1	11.40		7.40	
Per cent color removed		79.60		86.80	

FURTHER ILLUSTRATION OF THE USE OF THE TINTOMETER

For some years the writer had been experimenting upon a decolorizing black, which, it was believed, might be useful as an auxiliary to bone-black filtration. The decolorizing power of this black had frequently been determined and used for refining purposes without the subsequent aid of bone-black, but certain technical difficulties soon indicated that its value would be restricted to auxiliary work with char.

The figures submitted below are typical of several groups of experiments made in decolorization by means of this black (which will be designated by D. B.), by itself and also supplemented by bone-black.

Experiment I.—A raw sugar solution was prepared by defecation in the usual manner and made to a density of 55° Brix. The liquor thus obtained gave the following color analysis:

	Solution A	
		Per cent
Yellow	77.00	86.00
Red	11.50	12.80
Blue	1.00	1.20

Decolorized by D. B., color analysis gave:

	Solution B	
		Per cent
Yellow	38.00	88.30
Red	5.00	11.60
Blue	0.05	0.10

Total color removed by aid of D. B., 51.5 per cent.

Standard filtrations, as described under "comparative filtration," were then made upon equal volumes of solutions A and B and the resulting filtrates tintometrically examined.

	Filtrate from A		Filtrate from B	
		Per cent		Per cent
Yellow	2.70	81.8	2.20	81.5
Red	0.60	18.2	0.50	18.5
	3.30		2.70	
Color removed from original liquor		96.3		97.0

Apparently the effective work D. B. had done on Solution A, producing Solution B, was almost entirely lost after having passed through the bone-black. This result considered in connection with Experiment II incidently demonstrated the value of D. B. as a bone-black auxiliary, and also showed that exhaustion of the char is much slower when acting upon D. B. treated sugar liquors; in other words, either less bone-black is necessary to obtain a definite total decolorization, or, what amounts to the same thing, a greater volume of liquor may be put through a unit weight of char. This Experiment II amply demonstrates.

Experiment II.—A raw sugar solution was prepared as in Experiment I, and on color analysis gave:

	Solution C	
		Per cent
Yellow	45.50	79.00
Red	11.50	20.00
Blue	0.54	1.00
	57.54	

After decolorization by D. B. the color was:

	Solution D	
		Per cent
Yellow	18.00	86.10
Red	2.80	13.30
Blue	0.10	0.50
	20.90	

Color removed 63.6 per cent.

A unit volume of solution C was then filtered through 100 per cent bone-black on total solids in the solution.

A unit volume of solution D was simultaneously filtered through 66 per cent of bone-black on total solids in the solution.

The resulting filtrated from solutions C and D were color analyzed and gave:

	Solution C		Solution D	
		Per cent		Per cent
Yellow	3.00	97.4	3.10	80.00
Red	0.78	20.6	0.78	20.00
Total color removed from		93.4		93.39

As a last word it may be added that though the tintometer may be operated in fairly uniform daylight with the results indicated, the observations require considerable care and must be carried out systematically by one person, or two in collaboration, if comparisons are to be the most trustworthy obtainable. It will be readily understood that the personal equation is a factor of some importance in these observations, and a trained eye is always desirable.

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